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DECEMBER 15, 1947
VOLUME 36, No. 25

# AMERICAN DYESTUFF REPORTER

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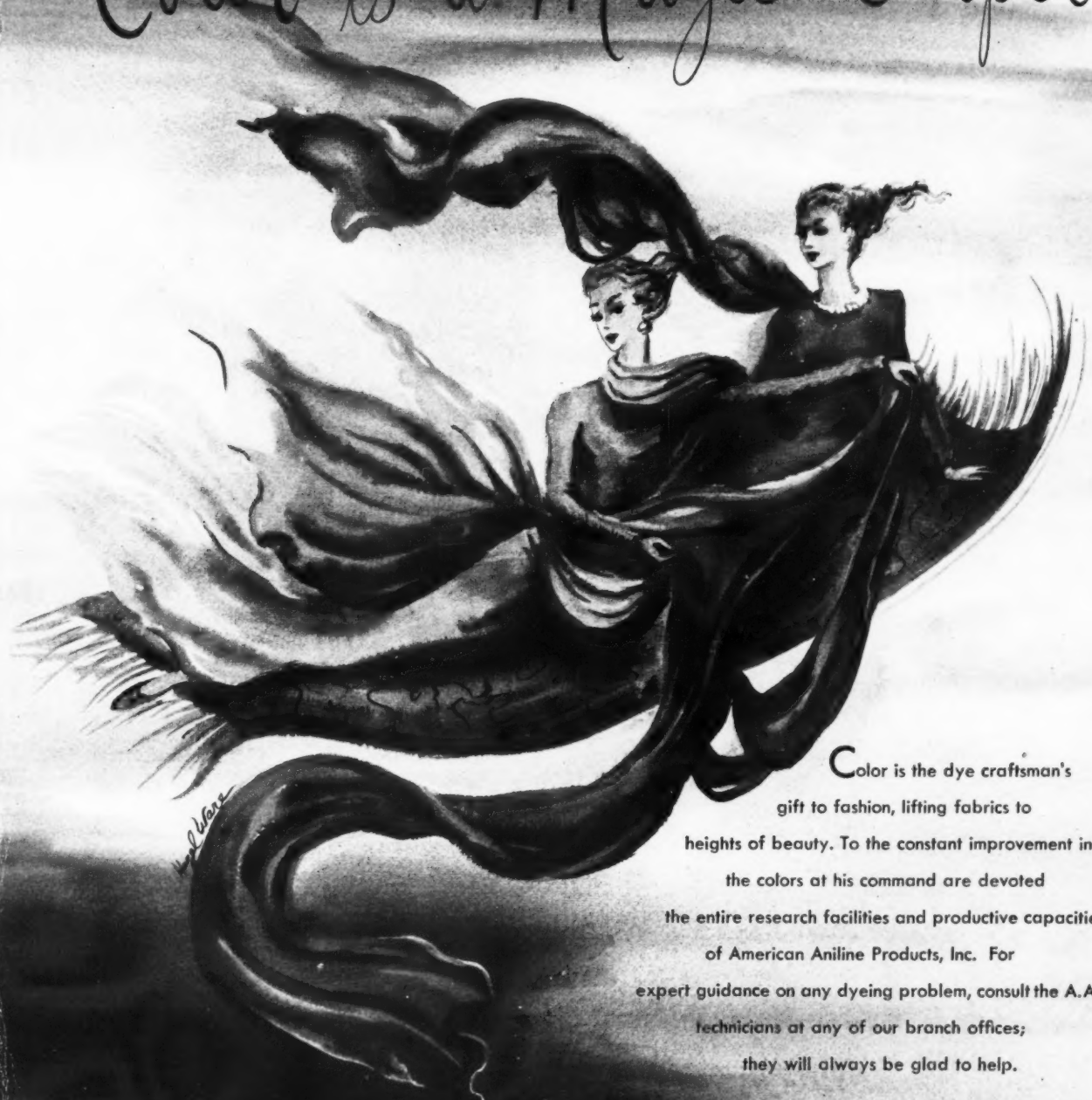

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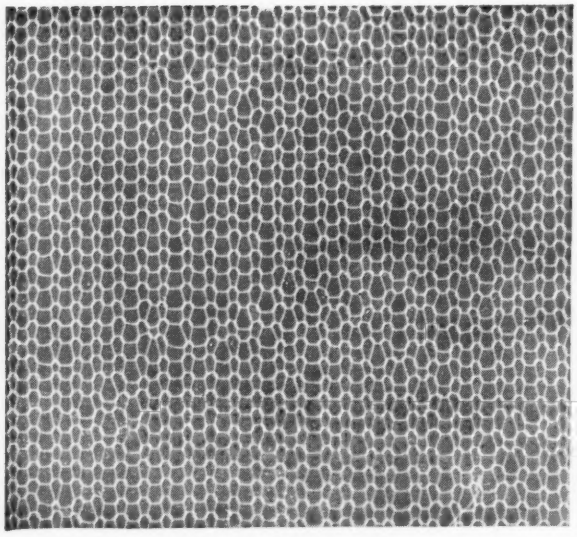

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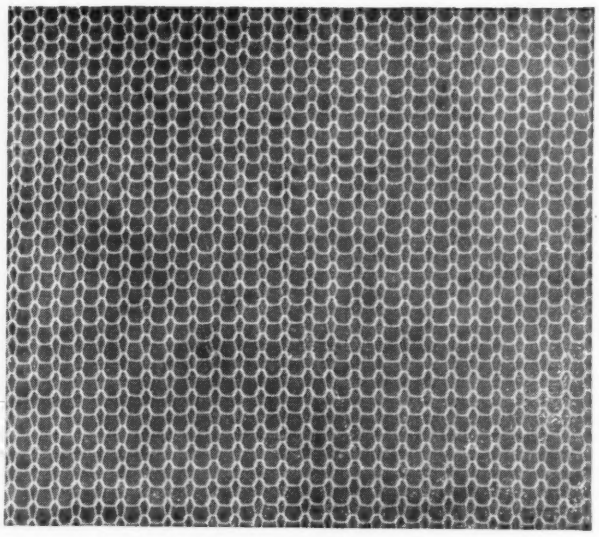

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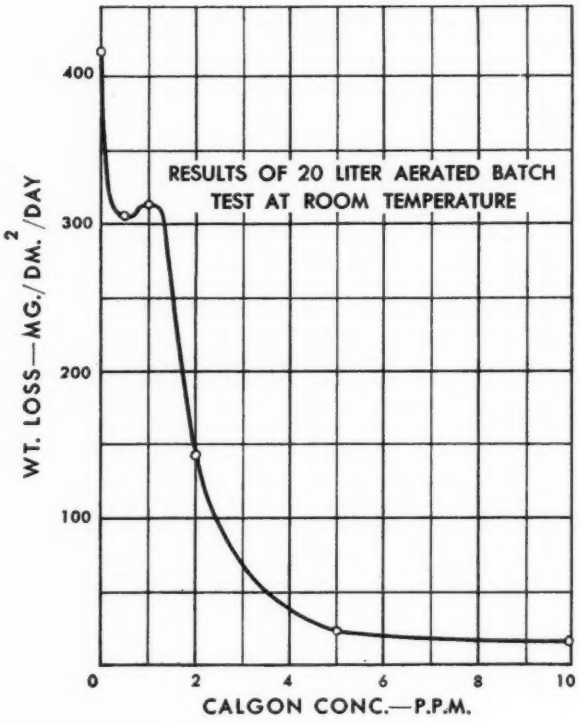


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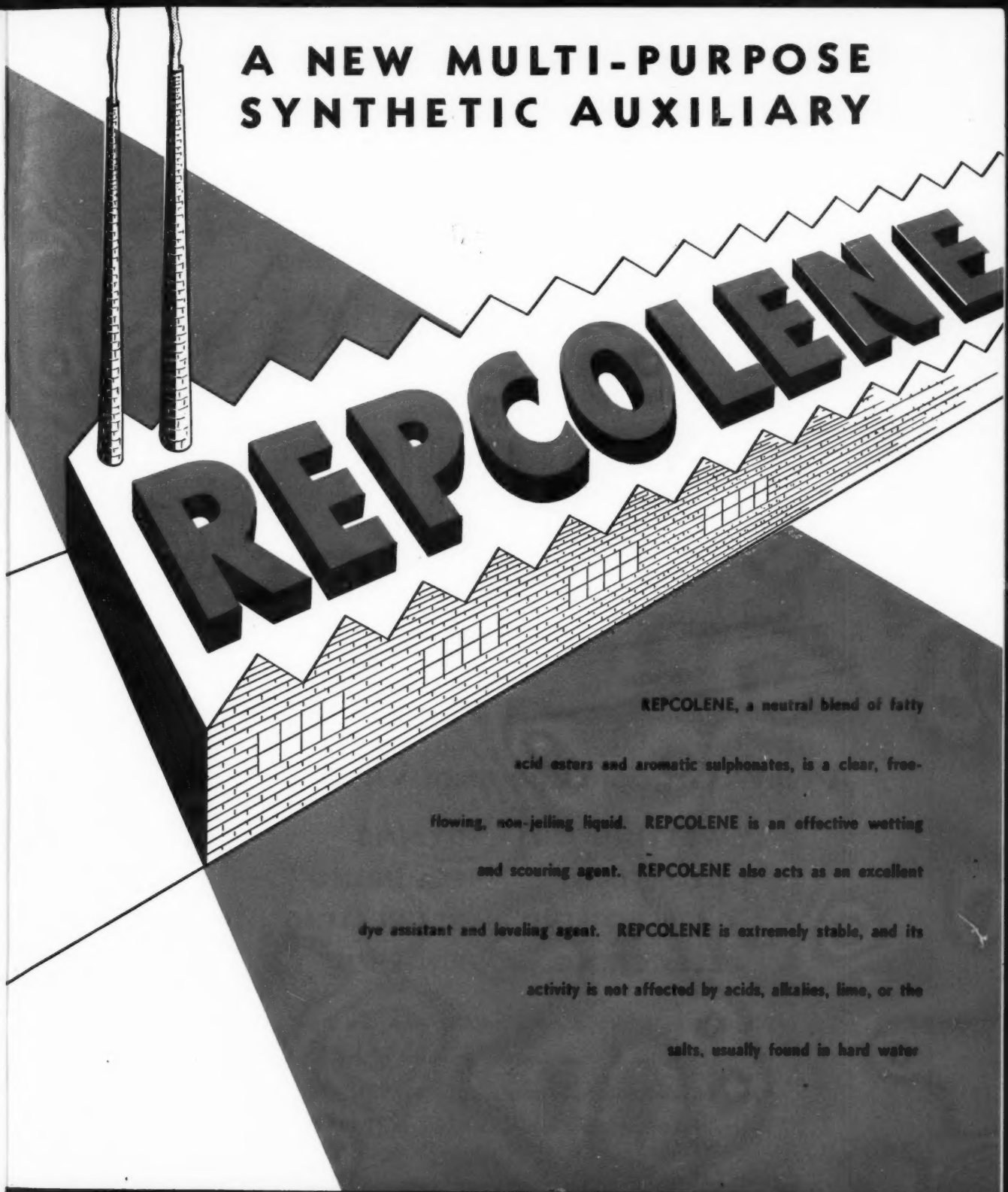


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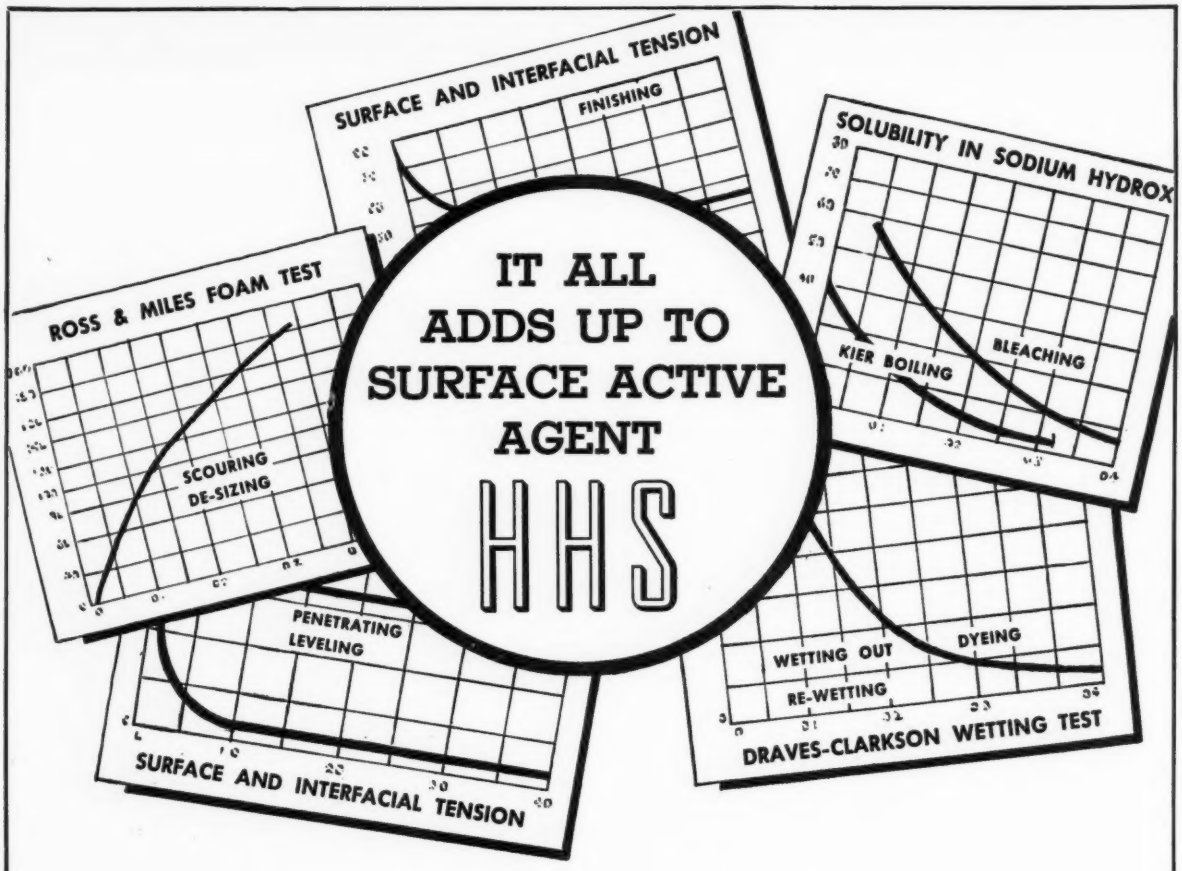


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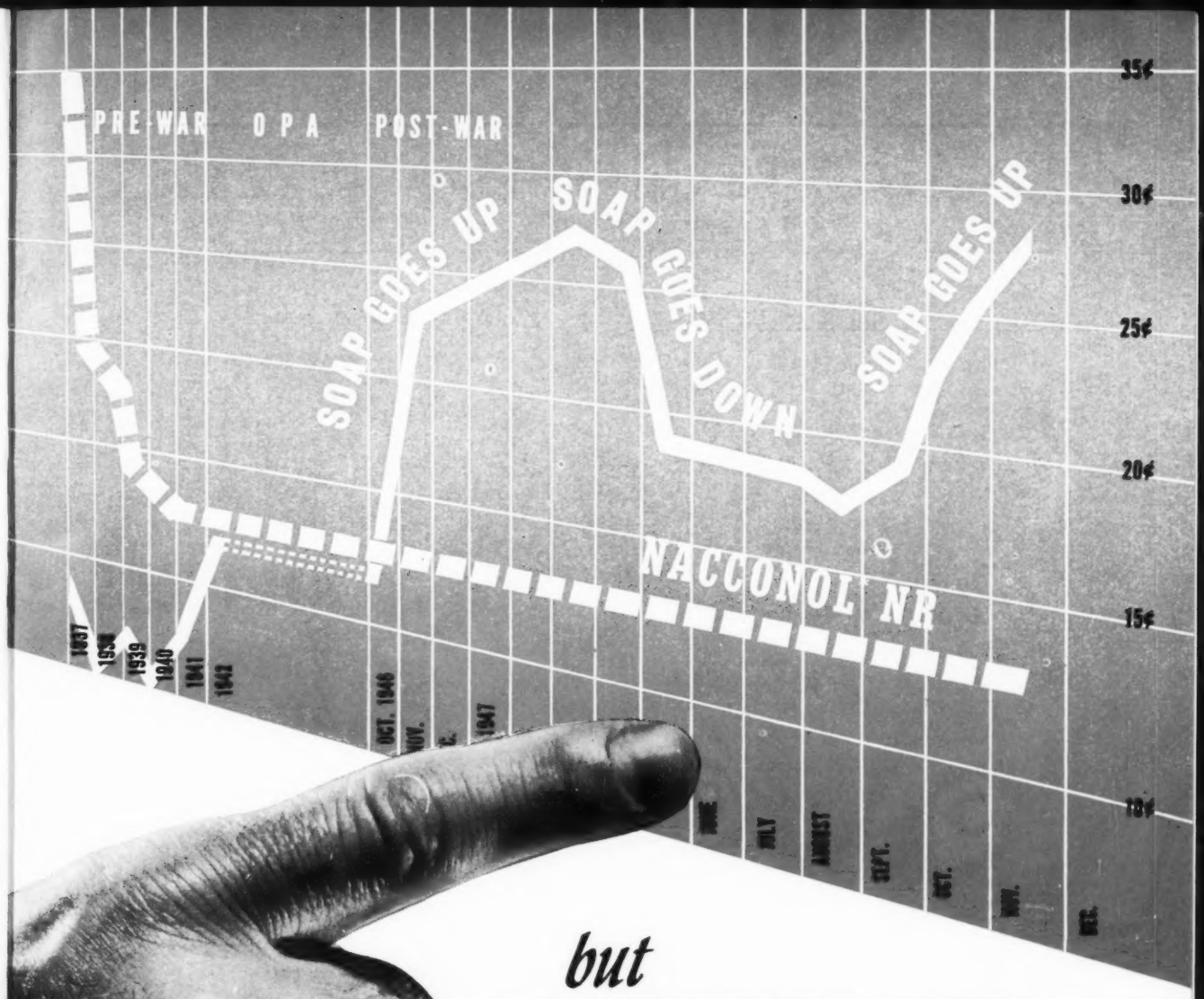


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**December 15, 1947**
**VOLUME 36 NUMBER 25**

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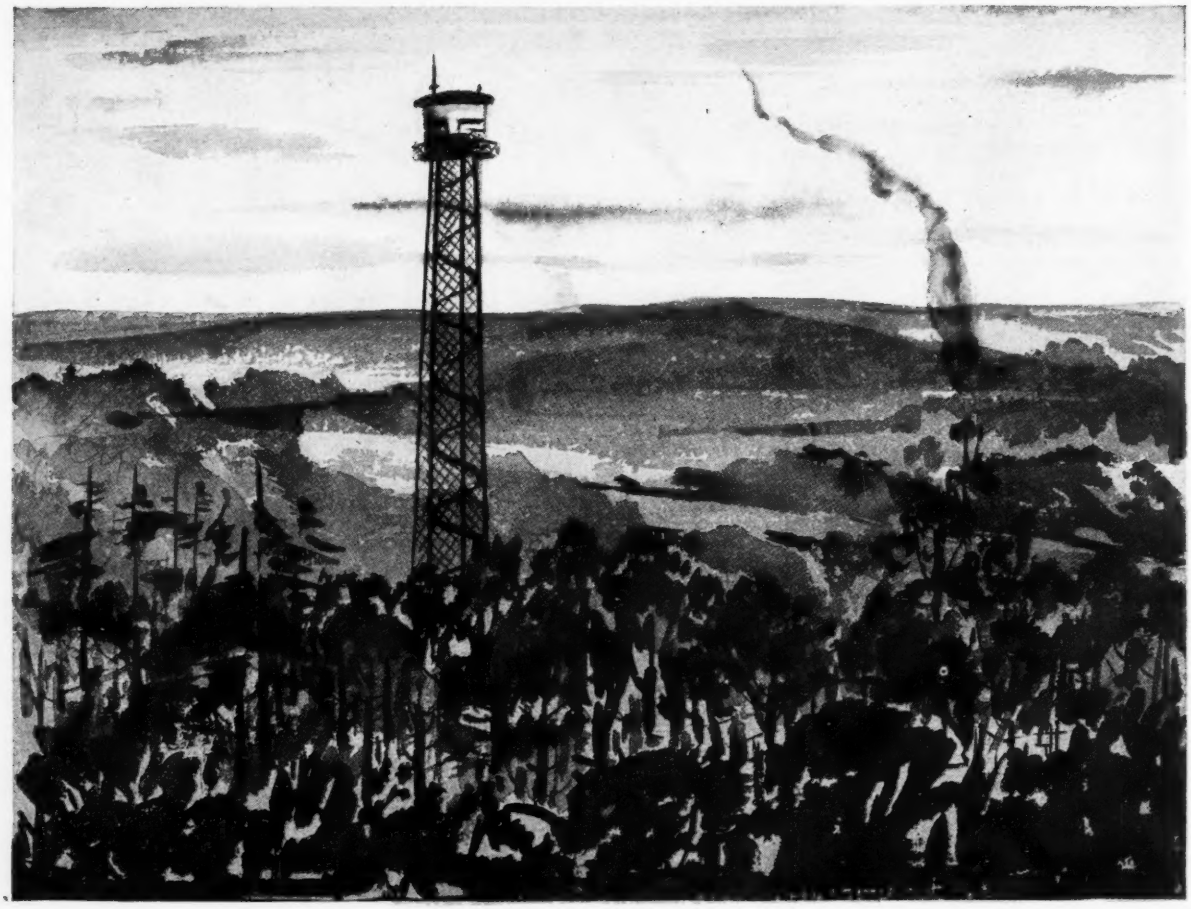

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DYESTUFF REPORTER

VOLUME 36 DECEMBER 15, 1947 NUMBER 25

# Shrinkage of Certain Cotton Woven Fabrics

## Caused by Various Laundering and Pressing Procedures

HAZEL M. FLETCHER and M. VIRGINIA JONES
*Textile Physicist* *Former Home Economist*
*Bureau of Human Nutrition and Home Economics, Agricultural Research Administration, United States Department of Agriculture*

CURRENT complaints of excessive shrinkage in cotton garments laundered by methods in which there is considerable tumbling point to the desirability of investigating the shrinkage of cotton fabrics when laundered by various procedures differing in the amount of mechanical agitation.

It is well know that mechanical action increases the felting of wool, and precautions are always exercised to keep rubbing and agitation at a minimum in laundering wool materials. A woven fabric of spun viscose rayon has been known to shrink as much as 30 percent after 40 washings in a tumbler wash wheel*(1)*.[1]

Studies on some phases of laundering of cotton materials have been made. One investigation *(2)* showed that the shrinkage of cotton fabrics caused by laundering by two home procedures and by two commercial launderies was similar. However, the amount of mechanical agitation in the different processes was not discussed. In another study *(3)* it was found that shrinkage in laundered cotton fabrics appeared to be overcome by pressure erected during ironing.

The study reported here was made in order to investigate the effect of mechanical agitation and the methods of pressing on shrinkage in cotton woven goods. The dimensional change of five commonly used cotton fabrics was determined when the materials were laundered by three different washing methods and three different pressing procedures.

### Materials

The five fabrics selected included two printed percales, a chambray, a printed crash, and a plaid gingham. All the materials were of plain weave, medium weight, and their yarns contained a moderate amount of twist. The weight per square yard ranged from 2.3 to 3.4 ounces; yarns per inch in warp from 61 to 85, in filling from 48 to 78; number of turns per inch (twist) in warp yarns from 21 to 27, in filling yarns from 21 to 29. No fabric contained any finishing materials which inhibited shrinkage. The amount of sizing, ranging from 2.9 to 5.3 percent, was not excessive. Only the chambray had been preshrunk mechanically.

Nine specimens, each 20 inches by 20 inches, were prepared of each fabric. Three 18-inch lengths were marked off accurately in both the warp and filling directions in accordance with the standard test method for preparation of specimens for shrinkage in laundering of cotton and linen woven fabrics *(4)*.

### Laundering Methods

Three specimens of each fabric comprised a set of test pieces. One set was laundered by a standard test method *(4*, p. 30), another by a commercial procedure, and a third by a home laundering method. All washings were repeated five successive times.

#### Standard Test Method

The cotton fabrics were laundered in the Bureau of Human Nutrition and Home Economics' laboratory according to the standard test method, using tap water of 12 parts per million hardness and a neutral high titer flake soap. The wash wheel was approximately 18 inches by 24 inches, and the average load was 3 pounds. The water level was 11 inches.

The actual washing and rinsing times were the same as those specified in the standard procedure, but the total time in the wash wheel was approximately one hour and ten minutes instead of one hour, because of the time required for the machine to drain and refill. A temperature range of 195° to 205° F. was used in order to eliminate hazards resulting from escaping steam at the 212° F. temperature.

#### Commercial Procedure

The specimens were washed in a commercial laundry with a regular load of soiled garments, according to the formula normally used for colored work.

The washing cylinder was 42 inches by 48 inches, and the average load of wash was 250 to 300 pounds. Softened water and neutral soap flakes were used in this procedure. The stock solution of soap contained 55 pounds of soap to 200 gallons of water.

The samples were given four 10-minute washes at a temperature of 120° F. To the first wash enough alkali was added to give a pH of approximately 11.0. In the next three washes enough of the stock solution of soap was used to make a "good running suds." The water level for these washes was 5 inches.

Five 3-minute rinses with a 10-inch water level followed. The first two rinses were at a temperature of 120° F., the next two at 100° F., and the last at 80° F. A commercial sour was used in the last rinse.

The samples were extracted for 10 minutes and then tumbled from 20 to 30 minutes until dry. The temperature in the tumbler at the beginning of the tumbling

[1] Numbers in parentheses refer to Literature Cited.

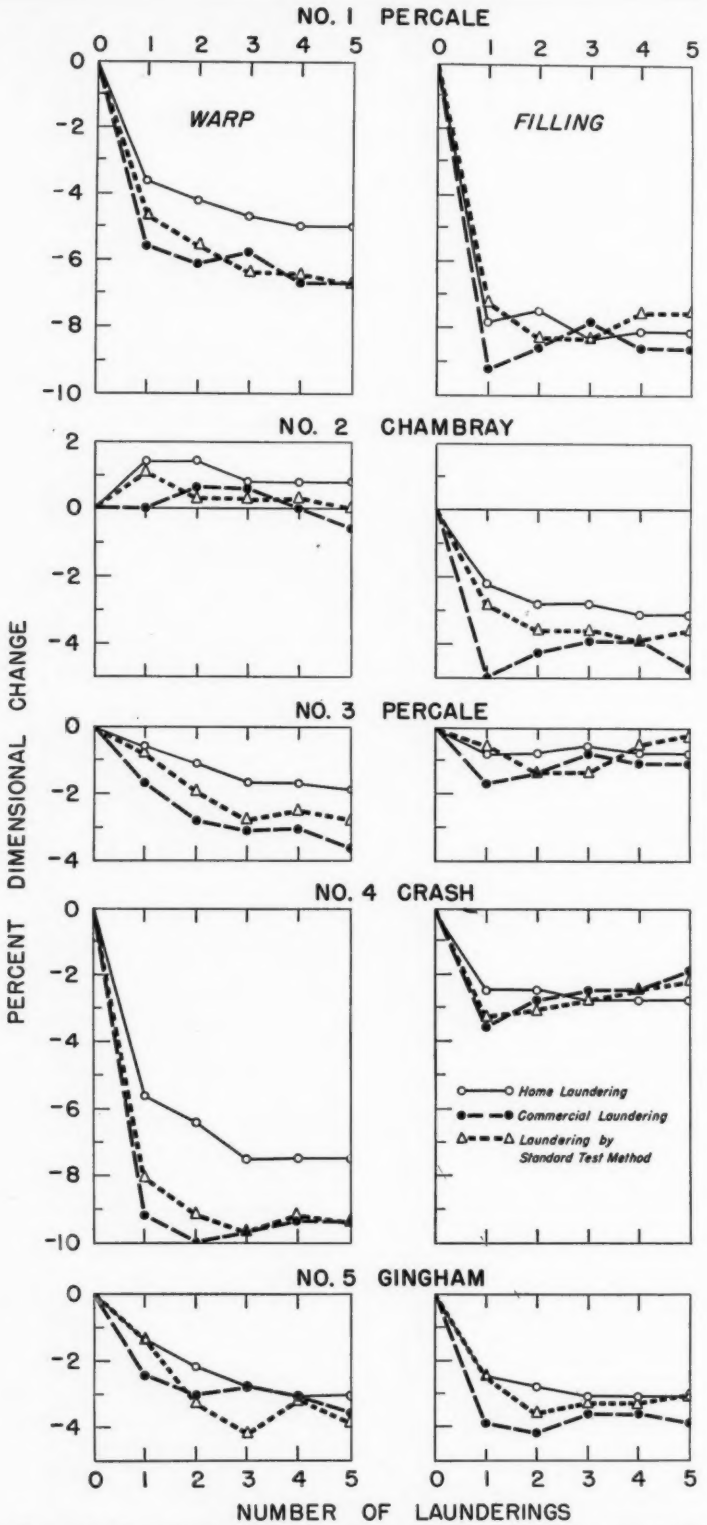


*Figure 1. Dimensional change in cotton fabrics caused by home laundering, commercial laundering, and standard test method and pressing by standard method of hand laundering.*

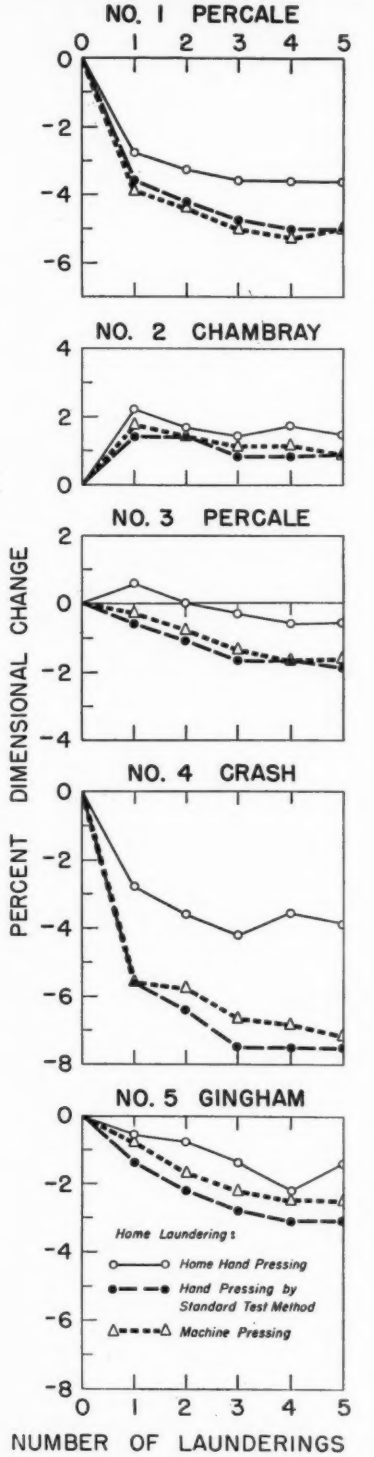


*Figure 2. The effect of three pressing methods on the lengthwise dimensional change in cotton fabrics caused by home laundering.*

period was 120° F. and at the end of the period 220° F.

### Home Procedure

The "home" laundering was done in the Bureau's laboratory using a vacuum-type electric home washing machine, with a built-in spinner for extracting. The washing procedure followed that commonly used for washing cotton garments and household linen in the home. When filled to the recommended water level, the machine held approximately 85 pounds of water. A load of 2 pounds was used for these washes. Tap water of 12 parts per million hardness and neutral soap flakes were used.

The machine was run for a few minutes to mix thoroughly 0.1 percent by weight of flake soap, 0.05 percent soda ash and water which gave a running suds before adding the test specimens. The samples were washed for 20 minutes at 120° F. They were given three 5-minute rinses. The first at 120° F. to which a 0.05 percent soda solution was added, the second at 120° F., and the third at 100° F. The samples were extracted in the spinning compartment of the machine for approximately 2 minutes after the wash and between rinses. The samples were spread out on screens to dry in the air at room temperature.

## Pressing Procedures

The dry specimens laundered by the three methods were laid out smoothly, without stretching, on a table, sprinkled uniformly, rolled, and allowed to stand for 5 minutes before pressing. All were pressed by the same operator by three different procedures. One of the three specimens of each material laundered by each method, was ironed by hand pressing according to the standard test method, another by flat bed machine pressing prescribed in the standard test, and the third by a modified home method of hand pressing.

According to the hand pressing method outlined by tht standard test the specimen was smoothed out on a padded ironing board and pressed by alternately raising and lowering the iron. The iron was not allowed to slide over the surface of the specimen. The temperature of the iron was between 275° and 300° F.

In the machine pressing the dampened specimen was laid out smoothly on the flat bed press and the head of the machine which had been pre-heated to 275° and 300° F. was lowered and allowed to remain in contact with the fabric until dry.

For the modified home method of pressing the temperature of the iron was the same as in the other two procedures. The samples were ironed first in the warp direction. The iron was always held with the side facing the operator and was pushed with moderate pressure across the fabric away from the presser. It was not slid back and forth. This same process was repeated in the filling direction. Each specimen was ironed in this manner until it was thoroughly dry, usually two times in each direction. With some of the heavier fabrics the process was repeated three or even four times.

All specimens were allowed to lie flat on screens at room temperature for one hour before measuring. They were then measured with a steel ruler at three points in the warp and filling directions.

## Results

The shrinkage caused in the cotton fabrics by the standard test procedures and the commercial processes was similar in amount. Commercial laundering usually caused slightly more shrinkage than laundering by the test procedure and both of these produced substantially greater shrinkage, especially in the warp direction, than was caused by home laundering. The amounts of shrinkage in five successive launderings by the three procedures and pressing by the hand method described in the accepted test procedure are shown in figure 1. The data reveal that the shrinkage in the home method for the fourth and fifth launderings was from 0.3 to 2.0 percent less than in the other methods.

The three pressing procedures resulted in differences in shrinkage. There was only slightly greater shrinkage with hand pressing when the iron was alternately picked up and put down than with machine pressing with the flat bed press. Hand pressing by the "home" method as previously described resulted in considerably less shrinkage than either of the others. The results for warp shrinkage of fabrics in home laundering and pressing by the three procedures are shown in figure 2. Shrinkage with the home hand ironing was in some instances as much as 3.5 percent less than with machine or hand pressing prescribed in the standard test procedure.

In general the greatest shrinkage occurred in the first wash, further shrinkage occurred in the second and third wash. Beyond that, little change took place. All the fabrics shrank both in the warp and filling except the chambray which had been preshrunk mechanically. This material shrank as much as any of the others in the filling direction but stretched slightly in the warp direction with all procedures of washing.

## Conclusion

Results of this study show that mechanical action does affect the amount a woven cotton fabric shrinks. In home laundering where the specimens were subjected to mild mechanical action in the washing and spinning, the shrinkage was consistently less, irrespective of the method of pressing, than when the fabrics were subjected to mechanical action both in the wash wheel and in the tumbler during drying.

The standard test procedure for determining shrinkage in cotton and linen woven fabrics is satisfactory in predicting the shrinkage caused by commercial laundering.

Maximum shrinkage, which is not produced until after the third laundering, can be measured by the standard test method if it is repeated successively for at least three times. Multiple tests of four or five launderings of cotton fabrics would be adequate for predicting shrinkage in cotton woven goods.

The method of pressing, as well as the washing method, affects the amount of shrinkage. A procedure in which tension is exerted along the warp and filling of the fabric results in considerably less shrinkage than a procedure in which no tension is exerted.

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(1) "The Problem of Fabric Shrinkage"—F. Bonnet, Rayon Textile Monthly 27, 403 (1946).

(2) "The Effects of Laundry Methods on Cotton Fabrics"—Adella Eppel Ginter, Margaret Shadduck, Margaret Partlow, and Thelma Pearson, J. Home Econ. 29, 319-326 (1937).

(3) "The Durability of White and Colored Cotton Materials"—Adella Eppel Ginter and Doris Rhodes. In Missouri Agr. Expt. Sta. Bul. 300, 83 (1931). Abs. in Expt. Sta. Rec. 66, 394 (1932).

(4) "Textiles—Testing and Reporting." Ed. 4—Commercial Standard CS59-44. National Bureau of Standards (1944).


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## • Directory of Commercial Laboratories

A complete listing of commercial and university testing and research laboratories throughout the country, together with indications of the type of commodities tested, has been compiled by the National Bureau of Standards. This pamphlet is now available from the Government Printing Office as NBS Miscellaneous Publication M187 entitled *Directory of Commercial and College Laboratories.*

Information is given concerning 220 commercial laboratories, with 80 branches or offices, and 189 college laboratories used for research and testing as well as instruction. Listings are arranged both geographically and alphabetically to facilitate the ready location of any laboratory. *Miscellaneous Publication M187 may be obtained only from the Superintendent of Documents, Washington 25, D. C., at 30 cents per copy.*

# TRADE NOTES • NEW PRODUCTS

## • Alco Southern Distributor

Alco Oil & Chemical Corporation have appointed the Wica Company, Inc., as Southern Distributor for their complete line of textile chemicals. The Wica Company offers complete services to textile mills throughout the South.

*William C. Caldwell, Jr.*

William C. Caldwell, Jr. organized Wica Company September 1, 1946, with offices, laboratory and warehousing facilities in Charlotte, North Carolina. William D. Van Derburgh, well known in southern textile circles, joined Wica Company, Inc., as Vice-President.

## • New Bedford Textile Institute Fiftieth Anniversary

At a recent meeting of the N.B.T.I. Alumni Association at Boston, a committee of prominent mill men made plans for the observance of the Fiftieth Anniversary of the opening of this Textile School. The occasion is to be observed in May of 1948. The date of this event will be announced later.

During the passing of years and the many changes of addresses, the graduates of this school who have not recently received Association notices would do well to send present day addresses to any of the officers:

Edmund Dupre, Corresponding Secretary
N.B.T.I.
New Bedford, Mass.

Howard Bates, Recording Secretary
53 Chestnut Street
New Bedford, Mass.

Francis Tripp
N.B.T.I.
New Bedford, Mass.

Harold Sturtevant, Vice-President
44 Brookside Road
Cranston, Rhode Island

John H. Grady, President
35 Hartford Street
Boston, Mass.

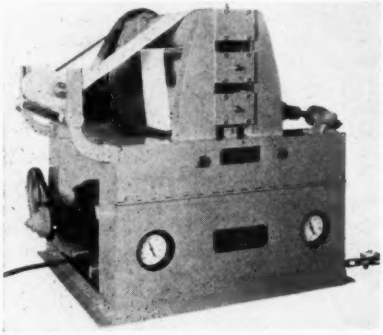
## • Asst. Director of Research Fabric Research Laboratories

Dr. Milton Platt has been appointed Assistant Director of Research of the Fabric Research Laboratories, Inc., 665 Boylston St., Boston, Massachusetts. Dr. Platt received his Sc.D. from Massachusetts Institute of Technology in 1946 in Structural Engineering and the Mechanics of Materials. During his graduate work at M.I.T. Dr. Platt did considerable research on various war-time programs on the mechanics of materials.

Dr. Platt joined Fabric Research Laboratories in August, 1946 and has been working on the application of stress-analysis techniques to textile problems as a part of the broad program of Fabric Research Laboratories in the application of engineering principles to textile, plastics, paper, and other non-rigid materials.

## • Butterworth Laboratory Padder

It is reported that H. W. Butterworth & Sons Company has noticed an increasing demand for all types of laboratory equipment. At the present time, the demand for the Butterworth Laboratory Padder is so great that the company is putting this machine through their shops in lots of 40.



*Butterworth Laboratory Padder, one of many laboratory machines being built by Butterworth for experimental work in textile mills.*

The industry purchased more than 100 of these laboratory padders in 1947.

The Butterworth Laboratory Padder has every facility of the larger dye house padder. Colors are matched and new formulas tested without the cost of an entire lot and without tieing up production machines. Pressures are perfectly controlled, assuring exact matches of shade on succeeding samples. Cloth is delivered from padder with minimum of moisture, thus speeding up the drying operation. The Butterworth Laboratory Padder has a friction let-off at entering side and slip belt winder at delivery side.

Among other laboratory machines being built by Butterworth are printing machines, agers, jigs, calenders, dryers, tenters, vat steamers and washers.

## • Southeastern Headquarters, Votator

L. N. Harrison, Southeastern District Manager of the Votator Division of The Girdler Corporation, has moved his headquarters from Charlotte, N. C. to 505 Forsyth Building, Atlanta, Ga., it is announced. The change was made November 1.

## • Booklet on Clorafin

Information on many new applications for Clorafin, Hercules Powder Company's chlorinated paraffin, is included in a new booklet which also covers revised information on the use of Clorafin in flame-proof and weather-proof textile coatings.

It is stated that considerable interest has been evidenced in the use of Clorafin 42 as a low cost plasticizer in pigmented vinyl coatings for cloth and metal and as a plasticizer for vinyl plastics. The new booklet includes the results of tests of vinyl coatings and vinyl plastics containing Clorafin.

Also discussed in the book are the general properties of the various types of Clorafins. An appendix to the booklet lists all trade names mentioned in the text and includes the chemical identity of these trade names, together with their owners.

## • Bulletin on Dyeometer

The versatility and convenience of the Dyeometer, an instrument used in conjunction with a photometric measuring

## • Joins Onyx

*Dr. David M. Musser, who joined the staff of the Onyx Oil & Chemical Co. on November 3, 1947, as head of the Textile Research and Development Division of the laboratories of this firm.*

*He is a graduate in chemistry of Pennsylvania State College. He holds a Master's degree from Georgia School of Technology. In 1937, he received his Ph.D. from the University of Wisconsin.*

*Following his doctorate, Dr. Musser was an Industrial Fellow at Mellon Institute, Pittsburgh, for five years, where he carried out research on a project of the Cotton Research Foundation. From 1942 to 1946 he was Senior Chemist in the research laboratories of Pacific Mills, and until acceptance of his new activity with the Onyx Oil & Chemical Co., was Senior Chemist in charge of research on textile finishing treatments with Deering Milliken & Co., Inc. Dr. Musser has been granted several patents in textile technology and has published numerous articles in various professional journals.*

*It is stated that Dr. Musser's extensive experience in the fields of textile finishing agents and textile processing, in the development of dyeing and other textile auxiliaries, and in the general chemistry and technology of natural and synthetic fibers will not only be available to Onyx customers, but will materially accelerate the progress of textile research in the Onyx laboratories.*

device, has been demonstrated in a series of studies involving dyes and textiles. These investigations have been carried out and reported on by the Research Staff of the Calco Chemical Division, American Cyanamid Company, Bound Brook, New Jersey.

The Dyeometer, described in detail in a recent Calco Technical Bulletin, gives data on change in the strength and color of a dyebath during the course of a dyeing. Since the most important factor to be considered in the dyeing process is speed, knowledge of the rate at which dye transfers to the fiber from the dyebath is essential to an understanding of the dyeing process.

Investigations to establish a comparison of the dyeing properties of various rayons and to study some of the dyeing conditions in the dyeing of rayon, were made by G. L. Royer, H. R. McCleary and J. M. A. de Bruyne, of the Research staff of the Calco Chemical Division, American Cyanamid Company. The result of these studies was presented by Dr. Royer in a paper before the Manchester Section of the Society of Dyers and Colourists, in England.

The paper, "Studies on Rayon Dyeing: Application of the Dyeometer", has been reprinted as Calco Technical Bulletin No. 795. Copies may be obtained from the Calco representative or by mailing your request to the Advertising Department, Calco Chemical Division, American Cyanamid Company, Bound Brook, New Jersey.

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## • Delegate to U. N. Conference

Dr. Elvin H. Killheffer has been appointed a member of the United States delegation to the United Nations Conference on Trade and Employment, which began in Havana on November 21st.

This Conference is to consider and improve the Geneva Draft of a Charter for an International Trade Organization.

"Prof. Glugg has a theory that if he isolates everything but the common cold germ, what's left has got to be it"

GARDNER REA

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## • National Inventors Council

A circular outlining the organization and functions of the National Inventors Council, comprised of 16 nationally known scientists, inventors, and industrialists, was released recently by the Office of Technical Services, Department of Commerce. Dr. Charles F. Kettering, retired vice-president in charge of research, General Motors Corporation, is chairman of the Council.

The circular describes the Council as a "clearing house for inventions and ideas which will assist the Army, Navy, and other public agencies in solving technical problems affecting the national defense and welfare."

To this end, the Council encourages private citizens to submit inventions and ideas which they believe will contribute to national defense and welfare. When in the judgment of the Council a proposal appears to be of value to the armed forces, the Council refers it to the proper technical authorities in the agency concerned. The Army and Navy in turn furnish the Council with summary statements in urgent need of solution. The Council publicizes these statements among researchers and inventors throughout the country.

Copies of the circular and summary statements of technical problems confronting the Army and Navy are available from the Council on request. Communications should be addressed to National Inventors Council, Department of Commerce, Washington 25, D. C.

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## • President, Chicago Drug and Chemical Association

Gerald F. Pauley, general branch manager of Monsanto Chemical Company's Organic Chemicals Division sales office in Chicago, was elected president recently of the Chicago Drug and Chemical Association. He will take office January 1.

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## • Leeds & Northrup Building

Purchase of the two-story and basement building at 34 E. Logan Street, one block from its main plant, by Leeds & Northrup Company, manufacturers of electrical measuring instruments, automatic controls and heat-treating furnaces, has been announced by C. S. Redding, president of the 48-year-old firm.

The building was owned and occupied by the Automatic Temperature Control Company, Inc., which has consolidated, at a new location, operations previously carried on at this and two other locations.

The newly acquired property will add

"Nice voice, but he doesn't appear to be much of a musician!"

TOM HENDERSON

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approximately 11,000 sq. ft. of floor space to the 288,886 sq. ft. now in use at the Leeds & Northrup plant on Stenton Avenue. The purchase price was not revealed.

The building will be adapted as quickly as possible, Mr. Redding said, to increase the effectiveness of services rendered by the company.

## • Production Begins in New Calco Plant

The first production unit of the new Willow Island, West Virginia, plant, of the Calco Chemical Division, American Cyanamid Company, has been completed and put into operation. A second large unit is scheduled for operation in January 1948. All construction work and installation of equipment scheduled for the current plant construction program is expected to be completed by early spring 1948.

The management of the Willow Island Plant of the Calco Chemical Division, American Cyanamid Company, is conducted by D. H. W. Felch, Plant Manager, J. W. Dykes, Production Manager, and R. C. Lantz, Personnel Manager.

## • Vice-President, Textile Colors Division

Interchemical Corporation — Textile Colors Division, Fair Lawn, N. J., has announced the election of William B. DePass, Sales Manager, as Divisional Vice-President. Mr. DePass joined the Textile Colors Division (then Aridye Corporation) in 1939 as a Technical Representative. Later he became District Manager of the Southern District and earlier this year was appointed Sales Manager. Following his graduation from Clemson Textile School in 1933, Mr. DePass went with Southern Bleachery & Print Works and subsequently with Ware Shoals Manufacturing Company.

## • General Releases

General Dyestuff Corp., 435 Hudson Street, New York 14, N. Y., announces release of circulars describing the following products:

*Rapidogen* Golden Yellow R* (Patented)—a stable azoic preparation, soluble in caustic soda, which after printing and acid ageing, develops in the form of an insoluble pigment possessing good fastness properties. When printed on cotton or rayon fabrics it produces bright, golden yellow shades said to possess good fastness to washing, light and chlorine. It is applied in the usual manner for Rapidogen colors. The stability in the print paste is good and the shade develops completely in acid ageing. It is suitable for the printing of fabrics which are to be given an imitation seersucker effect as its shade is not affected by overprinting with caustic soda. Circular G-490.

*Acid Chrome Red BA-CF*—a straight chrome dyestuff which produces exceptionally brilliant shades of bluish red. It can be applied equally well by all chrome dyeing methods. The resultant shades are said to possess good fastness properties. The solubility is good and the level dyeing properties sufficient to make the product suitable for application on raw stock, slubbing, yarn and piece goods on all types of dyeing machines. It leaves effect threads of cotton, rayon or acetate clean. It is a non-dusting powder and suitable for material which is to be rubberized. Circular G-491.

*Indanthrene Red Violet RHA Paste Fine*—a finely dispersed paste standardized for printing. It is non-settling and grit-free and has all-round desirable properties for vat printing. It fixes readily on cotton or rayon. The resulting prints are said to possess very good fastness properties. Circular G-492.

*Algol* Yellow GCA Paste Fine*—a straight vat dye said to be of unsurpassed brightness of shade. It is stated that it is of extreme interest for the dyeing of yellow shades as well as for bright greens. It is suitable for the dyeing of cotton, rayon, linen and silk. On spun rayon-wool blended material good uniformity is obtained. Being in well dispersed form it can be applied by the pigment padding method, in package machine dyeing and all conventional dyeing methods. The fastness to washing and chlorine is said to be very good. It is stated that the light fastness of the straight yellow dye, depending upon the depth of shade, is fairly good to good and in green combinations the light fastness is very good. In addition to the Paste Fine brand, the following types are available:

*Algol Yellow GCA Double Paste Fine*—twice as strong as the single paste, otherwise the same.

*Algol Yellow GCWF Powder*—a dispersed powder equal to the double paste in strength; suitable for pigment application and package dyeing.

*Algol Yellow GCA Powder*—a concentrated undispersed powder, six times as strong as the single paste. Circular G-493.

*Wool Fast Blue BLA Extra CF*—an acid dyestuff which produces bright reddish blues said to possess good fastness to fulling and sea water and fairly good fastness to washing and light. Treatment with bichromate improves the fastness to washing, sea water and fulling. Since the shade is not affected to any extent by chroming, it is said to be well suited for shading and brightening of chrome blues. As it exhausts well from a neutral bath it is useful for the dyeing of mixed fabrics. It resists Rongalite and is consequently useful for the production of colored discharge work. It is sufficiently free from copper to be suitable for material which is to be rubberized. Circular G-494.

*Indanthrene Red Violet RHA Paste for Dyeing*—a straight vat dye specially standardized for dyeing purposes. The product is said to be particularly well suited for dyeing cotton and rayon piece goods according to the pigment pad process or for dyeing cotton and rayon yarns in skein or package form. It yields bright reddish shades of violet said to possess very good fastness to washing and to chlorine and good fastness to light in medium shades. It is best applied in reduced dyeing according to a special IN process and in pigment pad-jig dyeing by padding as usual and reducing at 160 to 170°F. Circular G-495.

* Reg. U. S. Pat. Off.

*Proceedings of the*

# American Association of Textile Chemists and Colorists

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DECEMBER 15, 1947

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# Studies in Soiling and Detergency*

By J. R. CLARK and V. B. HOLLAND
*Cannon Mills Co.*

***Presented by***
**PIEDMONT SECTION**

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## I. Introduction

FOR the past several years there has been considerable thought and discussion on the subject of detergency and its evaluation. Undoubtedly this has been brought about by the appearance, during that same period, of a very great number of synthetic detergents on the market. This condition has created a demand for a suitable standard method whereby the efficiency of these products might be evaluated. In answer to this demand several technical associations have organized reesarch committees for this purpose. Some praiseworthy work has come from these committees as well as from independent investigators, but to date no instrument, method or soil has been accepted as a standard for detergency evaluation.

A greater majority of the work has been on instrumentation and method, and in the writers' opinion, methods with excellent possibilities have been evolved. In spite of this, however, it seems that a minimum of effort has been expended in the consideration of a standard soil or soils. Therefore the prime purpose of this investigation is the development and study of a standarl soil and its relationship to certain aspects of detergency evaluation.

## II. Review of Literature

J. A. Woodhead et al (*11*) in attempting to develop a method to improve reproducibility in detergency tests brings out that the most important factor in the method described is the random selection, for each test, of many pieces of soiled fabric each made at different times. The investigators report that in terms of standard deviation, the reproducibility of a determination was found to be about ± 7 when results were expressed as units of black removed under the conditions described. Further, it is possible to determine with reasonable assurance differences between detergents which are greater than 6%. In this work the tests were run in any ordinary home-type washing machine. It is suggested that the method might be adapted to measure relative efficiencies of various types of washing machines as well as for evaluating detergents.

Vaughn et al (*10*) describes a method for preparing soiled cloth whereby a large portion of the soil solution is prepared in a small commercial-type washer. Indian Head Fabric, cut into yard squares and desized, was used.

The soil solution consisted of:

| | |
|---|---|
| 128 Liters | Stoddard Solvent |
| 1600 grams | Melted Crisco |
| 800 mls. | Lubricating Oil |
| 240 grams | Norit C (200 mesh) |

Norit C was added to one-half the other ingredients, the remainder of which was used as a rinse after the soiling in the washer of the muslin squares.

A new method for the laboratory determination of the detergent value of washing preparations is reported by Jaag (*6*) in which pieces of cloth soiled with India ink, with India ink and mineral oil and with India ink and vegetable oil were washed with various detergents. Different detergents behaved differently toward the various types of soil.

Van Zile (*9*) gives claim to a satisfactory soil formula consisting of carbon black, mineral oil and vegetable oil in $CCl_4$. The fabric is drawn through a soiling box and then into a chamber of baffles. This oblong chamber is heated by 10-kw. heaters which can be regulated in steps of 250 w. and air is drawn through it and discharged by means of a variable speed blower.

One publication (3) reports a test soil which is prepared by mixing (A) oil 30, lanolin 5, egg yolk 1, egg albumin 1 g. and (B) milk 250, cacao 20, starch 4 and sugar 10 g. B. is mixed cold, heated to boiling and diluted with 250 grams of water. One part of A is blended with 9 parts of diluted B. The mixture is applied with a viscose sponge to strips of mixed cotton and rayon fabric 20 cm. by 150 cm., the sizing having been removed from the cloth previously.

Kelly (*7*) says that the laboratories of his company uses for a standard soil a combination of Oildag, Nujol, and edible tallow dispersed in carbon tetrachloride. He considers the Launder-Ometer as probably the most widely approved instrument for the evaluation of all types of detergents and believes that it comes nearest to offering standardized results.

Bacon (*1*) describes a method for the preparation of a standard soiled cloth using:

| | |
|---|---|
| 60 grams | Germantown Lampblack |
| 180 grams | Crisco |
| 258 grams | Nujol |
| 120 grams | Wheat Starch (200 mesh) |

Dilute 10.3 grams of the above to 1000 ml. using $CCl_4$.

The Launder-Ometer is used in the washing procedure.

Holland and Petrea (*5*) employed a standard soil having the following composition:

| | |
|---|---|
| 89.0% | Stoddard Solvent |
| 2.5% | Vaseline |
| 2.5% | Paraffin Wax |
| 2.5% | Stearic Acid |
| 2.5% | Oleic Acid |
| 1.0% | Norit C |

In addition to the usual reflectivity measurements, they determined the actual amount of soil removed by weight and by extraction, the quantity of solvent soluble material remaining in the cloth after washing.

As a standard soil for cotton, Crowe and co-workers (*4*) used 6 grams of Oildag, 1.7 grams Wesson Oil and 3000 cc. $CCl_4$ as a carrier. For wool the standard

* Presented at Intersectional Contest, National Convention, Chicago, Ill., October 25, 1947.

soil was 0.5 grams Lampblack, 7.5 grams Edible Tallow, 25.0 grams Nujol and 4000 cc. $CCl_4$.

Snell (*8*) uses a standard soil composed of a carbon black-oil mixture which is diluted with carbon tetrachloride. The Launder-Ometer is not recommended in that its reproducibility is poor. For this reason a household washer or small commercial wheel is preferred in this report.

In addition to the usual measurements, the same investigator (*8*) attempts to determine the dispersing power by shaking coated burnt umber with a given detergent solution and ascertaining the extent of the suspension.

## III. Development of a Standard Soil

Standard Soils now in common usage all have some outstanding fault which in a majority of cases outweighs the virtues of a particular solution.

In order to fully evaluate the essential qualities of any given detergent for textile purposes, it is necessary to have a standard soiled cloth which will contain a definte amount of oil or wax of a type as nearly inactive as possible. Many materials will not do for this reason, since their innate detergency values will tend to react unpredictably with the detergent being evaluated. Also necessary is a color agent, a portion of which can be removed by washing in order to give a check on reflectivity. Neither the oil nor the color agent should be too easily removed, as high concentrations of the detergent would cause results out of all proportion to its value. Another common fault to be avoided is the tendency of a soiled sample to become less washable in direct proportion to its age due to oxidation. Finally, the solution must coat the cloth uniformly as to color and to weight. Though not absolutely essential, the solvent used should not be too volatile, and should have some tendency toward holding the color agent in solution, or suspension, as the case might be.

### General Procedure and Method of Tabulation

The cloth used in this phase of experimentation was 64 x 64 sheeting, desized and bleached. Samples were cut 4" x 4" and overedged to prevent loss of threads in laundering.

Reflectivity measurements were made with a Hunter Multi-purpose Reflectometer, using the green filter (0.612) with the 60-AZ standard.

All extractions were run in soxhlet extractors for three hours using ethyl ether.

Results were calibrated as percentages as follows:

*Soil added*—Increase in weight after soiling against bone dry weight of unsoiled cloth.

*Soil removed*—Loss in weight after laundering against weight of soil added.

*Brightness Increase*—Amount of increase in percent between unwashed and washed sample.

*Percent Oil Remaining*—Amount of oil extracted in soxhlet against weight of soiled, unwashed cloth.

The formula selected, its preparation and method of application is given below. In the case of the various formulas tried and rejected, their formulas, preparation, application and general results obtained, are given either in the heading of their classification, or under "Results" after each formula.

Four duplicates were run in each series of tests, of which the best three selected as examples in each case.

Soil Formula:

0.25% Alkanol WXN (alkyl aryl sulfonate)
2. 0% Oildag
8. 0% Gulf 3F Fibre Spray Oil
89.75% Stoddard Solvent

*Soiling Procedure:*

A stainless steel tray 6" x 6" x 3", was constructed to hold the soiling solution. Samples are immersed in the solution by hand, held parallel to, but in no case touching, the bottom of the tray, and are agitated slightly for 15 seconds. Remove, drain, and strip excess solution from the cloth by drawing the thumb and forefinger of one hand lightly down the cloth in a parallel over-lapping pattern until the entire sample has been stripped. Place in a speed dryer (a whirlwind sample dryer was used) at room temperature for ten minutes. An alternate method is to place the sample on a wire rack with a bleached piece of sheeting both under and over it, and dry flat in a steam oven at 95-100° C. for 30 minutes. Unless a cloth is placed *over* as well as under the sample, the under-side will become lighter than the top side due to absorption by the sheeting. A reflectivity reading, front and back is then made on each sample. Following this the samples are dried to constant weight.

*Washing Procedure:*

Varying concentrations of detergents in distilled water as desired, are heated to approximately 160° F. and 200 ml. are placed with a soiled sample and 10-¼" steel balls in a Launder-Ometer jar. This is run at 160° F. for 45 minutes at 42 rpm. in the Launder-Ometer. The samples are then rinsed in 200 ml. of distilled water at 140° F., giving 50 double shakes by hand, and followed by two cold rinses of 200 ml. each of 50 double shakes. The soiled cloth is run through a wringer, and dried to constant weight. The percent soil laundered out is calculated and the reflectivity is again measured. Three hour extractions, as mentioned before, are then run; the oil extracted is dried and weighed and the percent oil is calculated. For more detailed information, the pH of each detergent solution was determined before laundering.

*Comment:*

Some differences in the amount of soil of a certain type which could be added to like samples arose. Probably the greatest factor affecting this was the moisture content of the sample. Although the proportion of soil removed remains fairly constant regardless of the amount of soil added, a cloth that would weigh exactly (within one or two percent, that is) as predicted is preferred. Possibly the best method would be to soil the cloth immediately after its first weighing while it is still in a bone dry condition. This should ensure fairly constant results. The addition of a wetting agent, Alkanol WXN for example, was found to increase the amount of soil added, and at the same time it helped to increase the evenness of the shading.

## Section A

### Aqueous Solutions—Starch or Gum Paste. Mineral Oil Emulsions—Pulverized Carbon Color Agents

*I. Procedure:*

Mix gum (or starch, as the case may be) and a small amount of water at room temperature into a thin paste. Heat remainder of water on steam bath to a minimum temperature of 95° C. Pour in gum, stirring rapidly until solution reaches maximum viscosity and begins to thin, then add the oil. Continue stirring until it is thoroughly emulsified. Pour into Mason Jar, add carbon and shake vigorously until carbon is evenly distributed.

*II. Preparation of Cloth:*

Submerge cloth in a beaker of the solution; allowing it to fully soak into the fibers. Slightly better results are obtained by applying solution with a paint brush. Run samples through a wringer, and dry flat on a cloth-covered wire rack in a steam oven for 30 minutes at 95-100° C.

*III. Formulas and General Results:*

Given individually on the following pages.

*IV. Comment:*

Solution #9 showed the only real promise, but a thorough check indicated its unsuitability. It was impossible to obtain a shade that would remain within a 5% variation between samples. The starch (or gum) carrier, being water soluble, washed out almost completely, even in small concentrations of detergents, and the carbon went with the starch into a soap emulsion.

*Solution #1:*

10.0% Starch (Clinton 60 F.)
1.0% Heavy Mineral Oil
1.5% Norit Carbon
88.5% Water

The solution was too thick to fully soak into the fibers, causing an undesirable stiffness, an uneven coating, and producing a light shade of grey out of all proportion to the heavy amount of carbon used. Spotting was caused by excessively large globules of oil and carbon, the result of mixing those ingredients previously to emulsifying oil.

*Solution #2:*

5.0% Starch (Clinton 60 F.)
3.0% Heavy Mineral Oil
0.5% Norit Carbon
91.5% Water

Results:

Approximately the same as in #1, though darker and a little more even, the sample was slightly stiff and spotty.

*Solution #3:*

4.0% Starch (Clinton 60 F.)
4.0% Heavy Mineral Oil
0.4% Norit Carbon
91.6% Water

Results:

Not enough starch to carry oil. Poor emulsion with excess of oil, and lumps of carbon on top of mixture. Sample blotted; too stiff, and of no value at all.

*Solution #4:*

4.0% Starch (Clinton 60 F.)
2.0% Heavy Mineral Oil
0.2% Norit Carbon
91.8% Water

Results:

Worse than #3. Virtually none of oil emulsified. Useless to even take a sample.

*Solution #5:*

6.0% Starch (Clinton 60 F.)
2.0% Heavy Mineral Oil
0.2% Norit Carbon
91.8% Water

Results:

Fairly even coating, but slightly stiff. Shade lighter than desired.

*Solution #6:*

5.0% Gum (Ace W)
3.0% Heavy Mineral Oil
0.5% Norit Carbon
91.5% Water

Results:

Solution much too thick. Did not coat sample evenly or thoroughly. Very low percentage of carbon adhered. Much too stiff.

*Solution #7:*

3.0% Gum (Ace W)
3.0% Heavy Mineral Oil
0.5 Norit Carbon
93.5% Water

Results:

Still slightly stiff and uneven. Slightly lighter than desired, and very spotty due to another abortive attempt to mix oil and carbon before emulsifying. Carbon tends to lump when introduced in manner first described, but is even less fully distributed when mixed in oil.

*Solution #8:*

2.0% Gum (Ace W)
3.0% Heavy Mineral Oil
0.5% Norit Carbon
94.5% Water

Results:

Slightly uneven and a bit light, otherwise fairly good.

*Solution #8 A:*

2.0% Gum (Ace W)
3.0% Heavy Mineral Oil
0.5% Norit Carbon
0.5% Norit Carbon (After Solution cooled)
94.0% Water

Results:

Very close to correct shade. Slightly uneven.

*Solution #9:*

3.0% Gum (Ace W)
4.0% Heavy Mineral Oil
1.0% Norit Carbon
2.0% Sodium Oleate
90.0% Water

Results:

Good shade, only very slightly uneven, not too stiff. (Soap gives a slightly better emulsion, but for accurate use of sample with detergents, its use is not recommended).

*Solution #10:*

1.5% Gum (Ace W)
1.5% Starch (Clinton 60 F.)
1.0% Norit Carbon
2.0% Sodium Oleate
4.0% Heavy Mineral Oil
90.0% Water

Results:

Fairly even, but a little dark.

*Solution #11:*

3.0% Gum (Ace W)
2.0% Wax (125° AMP)
4.0% Linseed Oil
0.2% Norit Carbon
90.8% Water

Results:

A graphite-like precipitate formed, which gave the sample an oily texture, but stained it only where the precipitate was rubbed on.

*Solution #12:*

0.5% Cation-active Softener
1.0% Borax
0.2% Bone Black
7.5% Heavy Mineral Oil
90.8% Water

Results:

Borax was added to rest of solution after it was mixed to increase the pH for better penetration. Solution was a poor emulsion made worse by the addition of borax. Not practical.

## Section B

### Stoddard Solvent Solutions—Petroleum Jelly or Heavy Mineral Oil Solutes—Pulverized Carbon Color Agents

*I. Procedure:*

Mix oil (or jelly) with the solvent. A slight amount of heat aids in dissolving the jelly. Put carbon in just prior to soiling sample as it will settle out of solution in a very short time.

*II. Preparation of Cloth:*

Place solution in Launder-Ometer jar (add carbon, as mentioned above) put in 10-¼" steel balls, and insert cloth Fasten jar into Launder-Ometer and run for 30 minutes at 42 RPM's. Remove sample, drain, and dry flat on a cloth-sovered wire rack in a steam oven for 30 minutes.

*III. Formulas and General Results:*

Given individually on the following pages.

*IV. Comment:*

In this group, Solution #5-A appeared to present the best qualifications. A detergency evaluation was run on this soil using the method previously explained. The brightness increase and the percent of soil removed was rather high for the low concentration of detergent used. A serious drawback to this type of soil, is the settling rate of the carbon. It is virtually impossible to keep the solution agitated enough to hold the carbon in suspension while inserting the sample into the Launder-Ometer. This results in heavy creasing and uneven soiling of the sample.

*Solution #1:*

5.0% Petroleum Jelly
0.5% Norit Carbon
94.5% Stoddard Solvent

Results:

This sample was run for 60 minutes. Was too dark, uneven, and carbon collected in the creases.

*Solution #2:*

7.5% Petroleum Jelly
0.2% Norit Carbon
92.3% Stoddard Solvent

Results:

Slightly lighter than #1, but with the same faults. Also run for 60 minutes.

*Solution #3:*

5.0% Heavy Mineral Oil
0.2% Bone Black
94.8% Stoddard Solvent

Results:

Very poor. Not enough oil to carry carbon. Carbon caked on cloth and on steel balls, leaving solution clear.

*Solution #4:*

4.0% Petroleum Jelly
0.1% Bone Black
95.9% Stoddard Solvent

Results:

Virtually the same as in #3. Not enough oil, but carbon did not cake quite as badly.

*Solution #5:*

7.5% Petroleum Jelly
0.2% Bone Black
92.3% Stoddard Solvent

Results:

Slightly darker than desired. Slightly uneven in coating. Approximately 10.26% reflectivity.

*Solution #5A:*

7.5% Heavy Mineral Oil
0.2% Bone Black
92.3% Stoddard Solvent

Results:

Gave a very desirable shade, but collection of carbon in creases remains. Approximately 26.08% reflectivity.

*Solution #6:*

7.5% Heavy Mineral Oil
0.2% Norit Carbon
92.3% Stoddard Solvent

Results:

Much too dark. Uneven shading.

*Solution #7:*

10.0% Paraffin—125° AMP
0.2% Norit Carbon
89.8% Stoddard Solvent

Results:

Slightly darker than #6, with identical faults.

*Solution #8:*

7.5% Heavy Mineral Oil
0.2% Activated Carbon
92.3% Stoddard Solvent

Results:

Much too dark. Very uneven.

## Section C

**Stoddard Solvent Solution*—Organic Oils or Petroleum Jelly Solutes—Oil Soluble Dyes, Color Agents**

*I. Procedure:*

Make a paste of the dye and a small amount of the oil. (If oleic acid is used, compound the paste with it before adding to oil). After smoothing out all lumps, add remainder of oil, and stir well. Pour into solvent and continue stirring until solution is homogeneous.

*II. Preparation of Cloth:*

Immerse sample in the solution for 30 seconds at room temperature. Remove, drain, and strip excess solution off by drawing sample between thumb and forefinger in a parallel overlapping pattern until the entire sample has been stripped. (It was found that a wringer tended to press the solution onto one side of the cloth more heavily than the other). Dry flat on a cloth placed on a rack in a steam oven at 95-100° C. for 30 minutes.

*III. Formulas and General Results:*

Given individually on the following pages.

*IV. Comment:*

Of these soils, it was decided to concentrate on an evaluation of solution #12-E, since it gave a very even shade, was easy to apply, and washed consistently. The Calco Oil Reds also seemed desirable at first, but several drawbacks were discovered. Foremost of these was a serious tendency to crock; the dye rubbing off, and removing a considerable amount of oil in the process.

In order to fully dissolve the dye used in Solution #12-E, it was necessary to use oleic acid, which has some detergency action in water. This one property lends some doubt to the advisability of using such a soil in detergency evaluation. Several detergency checks were made using this soil.

*Solution #1:*

5.0% Heavy Mineral Oil
0.5% Nigrosine
94.5% Stoddard

Results:

Fairly good, but dye settled out slowly, gave an uneven coating.

*Solution #2:*

10.0% Heavy Mineral Oil
0.5% Nigrosine SSB
89.5% Stoddard Solvent

Results:

Good, but too dark. Solution when filtered, gave a light violet filtrate, and left most of the dye on the paper. Not soluble enough to give the desired solution.

*Solution #3:*

0.5% Calco Oil Red, N-1700
0.5% Pine Oil
5.0% Oleic Acid
89.0% Stoddard Solvent

Results:

Gave a very even coating with a single short immersion. Red was used rather than black, because it was the best oil soluble dye available in the Laboratory at that time.

*Solution #4:*

0.5% Calco Oil Red N-1700
7.5% Petroleum Jelly
2.0% Oleic Acid
90.0% Stoddard

Results:

Slightly lighter than #3, but same good qualities.

*Solution #5:*

0.5% Calco Oil Red N-1700
7.5% Heavy Mineral Oil
82.0% Stoddard Solvent

Results:

About the same as #4. All Calco Reds *crock* badly.

*Solution #6:*

0.5% Calco Oil Black—B3804
7.5% Heavy Mineral Oil
92.0% Stoddard Solvent

Results:

Solvent precipitated dye out of solution at room temperature, and when heated also.

*Solution #6-A:*

0.5% Calco Oil Black—8603
7.5% Heavy Mineral Oil
92.0% Stoddard Solvent

Results:

Same as in #6.

*Solution #7:*

0.5% Calco Oil Black—B3804
10.0% Petroleum Jelly
89.5% Stoddard Solvent

Results:

Same as in #6 and #6-A.

*Solution #8:*

0.5% Calco Oil Black—8603
10.0% Heavy Mineral Oil
89.5% Carbon Tetrachloride

Results:

Fairly good. Still some insoluble matter suspended in solution, but dyed sample very evenly to a bluish-black shade. Solution must be kept tightly capped to prevent evaporation of carbon tetrachloride.

*Solution #8-A:*

0.5% Calco Oil Black—B3804
10.0% Heavy Mineral Oil
89.5% Carbon Tetrachloride

Results:

Fair, but with slightly more insoluble matter than in #8. Gave sample a greenish-black shade with some streaking from impurities.

*Solution #9:*

0.5% Calco Oil Black—B3804
10.0% Heavy Mineral Oil
89.5% Stoddard Solvent

Results:

Heated dye dissolved in oil to boiling, also heated solvent to boiling. Dye changed from greenish-black to a pinkish-violet shade and when added to solvent gave an orange-black solution with

* With the exception of solution #8 and #8-A where CCl, was used.

a black scum on the surface. The separating out of the black coloring matter made it impossible to get an even shade of the dye on a sample of cloth.

*Solution #10:*

0.5% du Pont Oil Black BG
10.0% Heavy Mineral Oil
89.5% Stoddard Solvent

Results:

Part of black element of dye formed on surface of solution as with the Calco Blacks. Apparently it is none too soluble in the oil and when solvent is added it lowers the solubility of the dye.

*Solution #11:*

0.5% Oil Black Jet Liquid
10.0% 3F Fibre Spray Oil
89.5% Stoddard Solvent

Results:

Only partially soluble. Darker elements refused to go into solution.

*Solution #12:*

0.5% General Powder Black #2671
10.0% 3F Fibre Spray Oil
89.5% Stoddard Solvent

Results:

Same as in #11.

*Solution #12-A:*

0.5% Calco Oil Black #B-3804
3.0% Oleic Acid
6.5% 3F Fibre Spray Oil
90.0% Stoddard Solvent

Results:

Few impurities undissolved. Dyed sample light gray shade—slightly spotted.

*Solution #12-B:*

0.5% Calco Oil Black #8603
3.0% Oleic Acid
6.5% 3F Fibre Spray Oil
90.0% Stoddard Solvent

Results:

Few impurities undissolved. Dyed sample blue-gray shade—slightly spotted.

*Solution #12-C:*

0.5% du Pont Oil Black BG
3.0% Oleic Acid
6.5% 3F Fibre Spray Oil
90.0% Stoddard Solvent

Results:

Few impurities undissolved. Dyed sample gray-brown shade—slightly spotted.

*Solution #12-D:*

0.5% General Oil Black #2671
3.0% Oleic Acid
6.5% 3F Fibre Spray Oil
90.0% Stoddard Solvent

Results:

Very few impurities remained difficult to put into solution, but very stable when thoroughly dissolved. *Do not heat* this dye in or out of solution. (A gummy mass is formed that is almost impossible to dissolve in Stoddard). Dyed sample blue-gray.

*Solution #12-E:*

0.5% General Oil Jet Liquid Black
3.0% Oleic Acid
6.5% 3F Fibre Spray Oil
90.0% Stoddard Solvent

Results:

Slightly lighter shade than 12-D. When washed reflectivity of sample rose from 13.90% to 18.92% an increase of 26.53% in brightness.

*Solution #12-F:*

0.5% National Oil Black #5115
3.0% Oleic Acid
6.5% 3F Fibre Spray Oil
90.0% Stoddard Solvent

Results:

Fairly soluble in oleic. Dissolves faster in pure oleic than in mixture of oleic and oil. Gives a darker shade than desired. Rather hard to remove all lumps and impurities, but once in solution, it stays in.

*Solution #13:*

0.5% National Oil Black #5115
10.0% 3F Fibre Spray Oil
89.5% Stoddard Solvent

Results:

Only slightly soluble. Not enough to give an even solution or suspension.

## Section D

**Stoddard Standard Solvent Solutions Containing Oil-dag (Colloidal Graphite in Oil), and Water Solutions Containing Aquadag (Colloidal Graphite in Water).**

*I. Procedure:*

In the case of the Oildag solutions, mix the Oildag with the oil before adding it to the solvent.

Simply mix the Aquadag with the correct amount of distilled water and stir rapidly.

Both colloids disperse evently and rapidly in their respective solutions.

*II. Preparation of Cloth:*

It was first attempted to soil the cloth by merely dipping the sample into a beaker of the solution. This, however, produced unsatisfactory results. Graphite settled in the creases, and in the case of the Aquadag, only poor permeability was obtained. Though not quite so even a sample is produced with the best soil of this group as is produced with the best of the Section C solutions, several of these soils are even enough for comparative checks within five percent.

A better method of soiling is to dip the sample in a metal tray of the solution, holding the cloth parallel to the bottom of the tray, and agitating it slightly. After 30 seconds immersion, remove and drain. Dry either in a speed dryer at room temperature 10 minutes, or flat on a cloth covered rack (with a cloth covering the sample) for 30 minutes at 95-100° C.

*III. Formulas and General Results:*

Given individually on the following pages.

*IV. Comment:*

Several of these solutions show promise. They are not too volatile, produce an even coating, wash out in a reasonable ratio to the detergency concentration, and only a small portion of the graphite settles out—that portion doing so slowly over a considerable number of hours, and it is easily returned to an homogenous state by a few vigorous shakes. Unlike the dye solutions, by using a thimble, the oil remaining in any of these samples may be removed without also removing the color agent (graphite, in this case). When extractions are run on dye soils, the dye is extracted in solution with the oil remaining—a rather undesirable property.

The Alkanol WXN, an oil soluble wetting agent, was used to obtain a faster soiling with a more even penetration. It increases the tendency of the graphite to settle, but not enough to alter the homogenity of the solution to any appreciable extent. It also has a slight detergency value, but checks show errors presumably due to this to be less than .75%.

*Solution #1:*

1.0% Oildag
7.0% 3F Fibre Spray Oil
92.0% Stoddard Solvent

Results:

Slightly lighter soil than desired. Some collection of graphite in creases, otherwise very even. Fast permeability. Tendency for some of the graphite to settle out overnight.

*Solution #2:*

1.0% Aquadag
99.0% Water

Results:

Rather uneven soiling probably caused by low permeability. Apparently more prone to settling out than Oildag. Shade good if evened out.

*Solution #3:*

2.0% Oildag
8.0% RF Fibre Spray Oil
90.0% Stoddard Solvent

Results:

Somewhat darker than #1. Overall shading uneven, and some creasing still evident. Applied solution to sample at room temperature and to other samples at 50° C. No appreciable difference in shading or amount of soil added.

*Solution #4:*

1.0% Oildag
2.0% Alkalol WXN
8.0% 3F Fibre Spray Oil
89.0% Stoddard Solvent

Results:

Slightly lighter than Solution #3. Gives a *very* even coat of soil. Too much Alkanol.

*Solution #5:*

2.0% Oildag
1.0% Alkanol WXN
8.0% 3F Fibre Spray Oil
89.0% Stoddard Solvent

Results:

Very good shade, but still too much Alkanol for use as a detergent evaluator.

*Solution #6:*

2. 0% Oildag
2.25% Alkanol WXN
8. 0% 3F Fibre Spray Oil
89.75% Stoddard Solvent

Results:

Excellent shade as in #5. 0.25% WXN sufficient as dispersing agent.

## Final Selection of Standard Soil

After careful analysis of the results obtained from all types of soils investigated, four soils were selected as having the most desirable characteristics. Samples were then soiled, washed and evaluated according to the method previously described.

For results, see Table #1.

Three of the four soils selected were from Section D of the experimental soils. The other was from Section C. This "C" type of soil was eliminated for several reasons, the most important of which was the fact that the dye remaining in the sample after washing was extracted along with the residual oil. Then too, the dyed samples tended to wash streakily, and unwashed samples tended to crock, or mark-off, badly on anything with which they happened to come in contact, and too much soil was removed in ratio to the detergent concentration.

Of the three Section D soils, number six was selected as something of a compromise. It would be desirable to eliminate all Alkanol as in number three, since almost any wetting agent has some detergent qualities, but without it a certain amount of streakiness appears caused by poor soil penetration. Solution number four while producing good results suffers loss of accuracy due to the large amount of Alkanol present. In a series of check experiments, it was determined that .25% Alkanol did not effect wash results appreciably. Consequently, number six was selected as the Standard Soil for further experimentation.

## IV. Wash Characteristics of Standard Soil as Affected by Age of Soiled Samples

The purpose of this phase of the investigation is to determine the effect, if any, of the conditions of storage of the soiled samples in regard to their wash characteristics. This work is similar to that of Bacon (*1*) in certain respects; however, the results of this paper may be compared with his only to a limited degree.

TABLE I
Soil Selection
Detergent—.25% Sodium Oleate

| Soil Solution | % Soil Added | % Soil Removed | % Brightness Increase | % Oil Remaining |
|---|---|---|---|---|
| # 3 | 8.40 | 66.94 | 22.94 | 3.35 |
| Section D.. | 9.01 | 66.90 | 23.72 | 3.64 |
| | 8.93 | 66.69 | 24.61 | 3.49 |
| | 8.57 | 67.30 | 23.53 | 3.41 |
| # 4 | 7.21 | 68.87 | 25.00 | 2.66 |
| Section D.. | 6.94 | 72.10 | 25.59 | 2.47 |
| | 6.78 | 73.28 | 24.31 | 2.33 |
| | 6.43 | 72.96 | 24.78 | 2.34 |
| # 6 | 6.54* | 68.16 | 21.28 | 2.74 |
| Section D.. | 6.52 | 63.55 | 19.41 | 2.60 |
| | 6.23* | 64.67 | 23.53 | 2.74 |
| | 6.43 | 64.44 | 22.16 | 2.62 |
| # 12-E | 10.28 | 93.47 | 27.35 | 1.33 |
| Section C.. | 11.22 | 94.06 | 26.96 | 1.36 |
| | 10.50 | 92.78 | 26.66 | 1.36 |
| | 10.41 | 93.83 | 25.69 | 1.41 |

* Rinsed twice in distilled water after soiling and drying to determine effect, if any, of Alkanol present in soil on soil adherence No apparent effect.

Two types of storage were used. One series was kept in a calcium chloride desiccator, and the other in an unsealed drawer. It is important to note that due to a rather long time interval between the evaluation of the two series, a wide difference in soiling method appeared. As improvements in applying the soil were proven practical, changes were made in the original procedure. The size of the samples was gradually increased over a period of time in an attempt to cut weighing error factors by increasing the sample area, and in consequence, the amount of soil present.

Samples of a series are directly comparable within the series, but allowances must be made for the two variations mentioned above when comparison between the series is desired.

### A. Sample Stored in a Desiccator

*PROCEDURE:*

Samples in this series were cut, soiled, and washed according to the procedure already given in Section III, Development of a Standard Soil under General Procedure and Method of Tabulation.

The samples of no age were washed immediately following soiling and weighing. The others were kept in a desiccator and were removed and washed at intervals as shown by figures 1, 2, & 3 under Results.

### B. Samples Aged in an Un-sealed Drawer

*PROCEDURE:*

Samples of bleached, but not finished 64 x 64 sheeting, over-edged and desized, and measuring approximately 4½ x 4½ inches were prepared. A sufficient number of samples were prepared to run evaluations for the desired detergent concentrations at intervals of no age to twelve weeks with a check for each, were bone-dried for two hours at 105° C., cooled in a desiccator, and weighed. They were then stapled together in groups of eight, with leaders on each end, with a wire stapler.

The Standard Soil Solution was padded onto the fabric samples by means of a shallow stainless steel tray and two rubber rolls having approximately 117 pounds pressure across the nip. Each group was run separately and with no further squeeze or treatment, were hung in a steam oven to dry for 30 minutes at 105° C.

From this point the samples were treated according to the method described previously under Part III except that they were put into a manila folder and placed in a drawer to age.

*RESULTS:*

Figures 1, 2 and 3 illustrate desiccator aging whereas figures 4, 5 and 6 portray the effect of aging in a drawer according to the conditions of this experiment. For a comparison of desiccator aging and drawer aging as regards the weight change of the soil due to aging see Table #2.

TABLE II
Weight Change Due to Aging of Soiled Samples
(Each figure represents an average of two samples)
(—) Indicates a loss in weight

| Age | Dessicator | Drawer |
|---|---|---|
| 3 days | +0.20% | .... |
| | —0.08% | .... |
| | —0.17% | .... |
| | —0.10% | .... |
| 1 week | +0.30% | —0.35% |
| | —0.13% | —0.41% |
| | —0.34% | —0.30% |
| | —0.20% | —0.03% |
| 2 weeks | —0.18% | —0.25% |
| | +0.02% | —0.40% |
| | —0.37% | —0.07% |
| | —0.04% | —0.05% |
| 4 weeks | —0.28% | —0.33% |
| | —0.22% | —0.35% |
| | —0.27% | —0.24% |
| | —0.26% | —0.20% |
| 6 weeks | —0.03% | .... |
| | —0.24% | .... |
| | —0.51% | .... |
| | —0.09% | .... |
| 8 weeks | —0.37% | —0.15% |
| | —0.52% | —0.41% |
| | —0.56% | —0.38% |
| | —0.34% | —0.48% |
| 12 weeks | —0.74% | —0.19% |
| | —0.56% | —0.69% |
| | —0.42% | —0.58% |
| | —0.37% | —0.44% |

*COMMENT:*

This experiment indicates only that a second baking (such as all the samples had—to determine the aging change—except the no age series) definitely makes the soil removal and brightness increase lower, and increases slightly the amount of oil remaining. Other than this the samples, regardless of age, are fairly well grouped—particularly in regards to

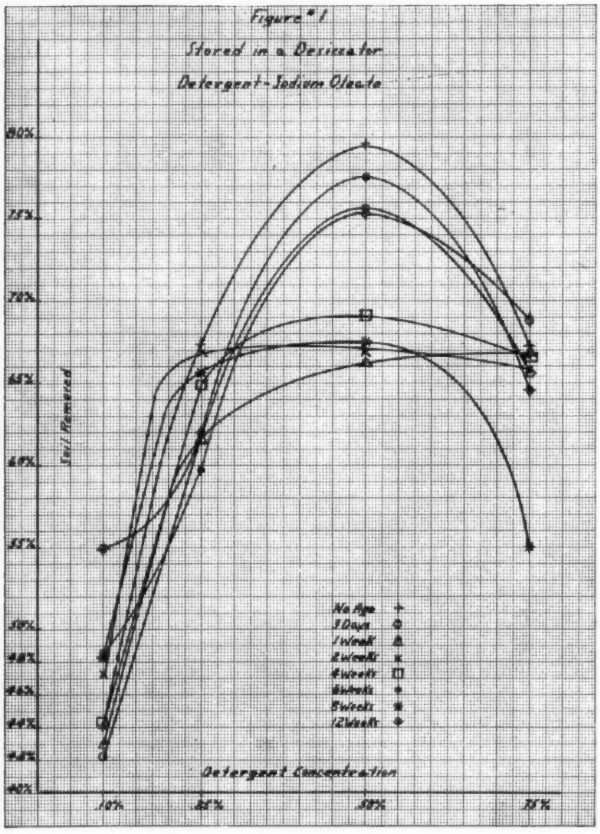


Figure #1
Stored in a Dessicator
Detergent - Sodium Oleate
Soil Removed
Detergent Concentration
No Age
3 Days
1 Week
2 Weeks
4 Weeks
6 Weeks
8 Weeks
12 Weeks

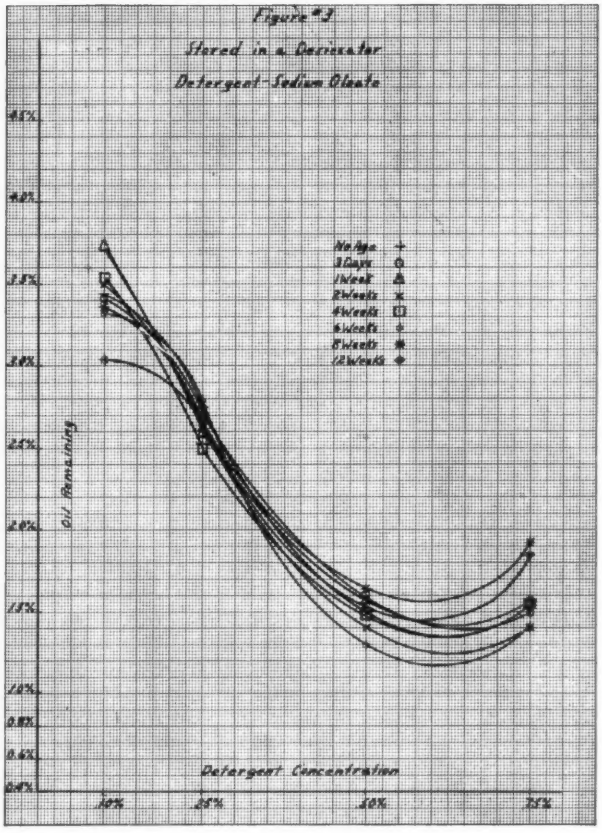


Figure #3
Stored in a Dessicator
Detergent - Sodium Oleate
Oil Remaining
Detergent Concentration
No Age
3 Days
1 Week
2 Weeks
4 Weeks
6 Weeks
8 Weeks
12 Weeks

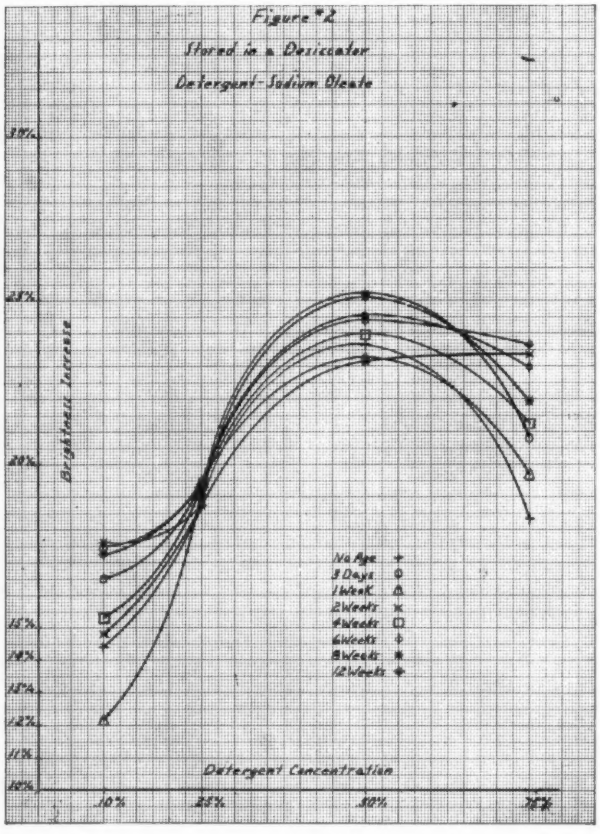


Figure #2
Stored in a Desiccator
Detergent - Sodium Oleate
Brightness Increase
Detergent Concentration
No Age
3 Days
1 Week
2 Weeks
4 Weeks
6 Weeks
8 Weeks
12 Weeks

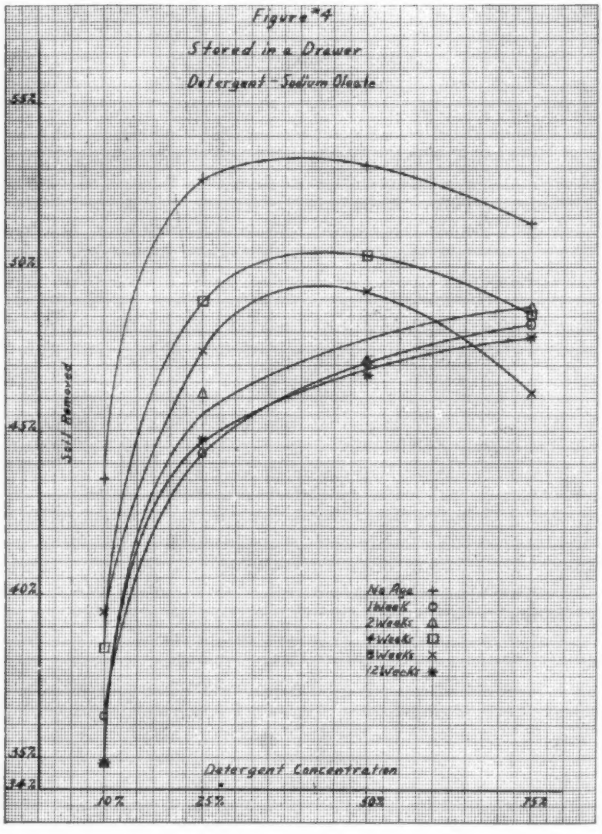


Figure #4
Stored in a Drawer
Detergent - Sodium Oleate
Soil Removed
Detergent Concentration
No Age
1 Week
2 Weeks
4 Weeks
8 Weeks
12 Weeks

Figure #5

Stored in a Drawer

Detergent-Sodium Oleate

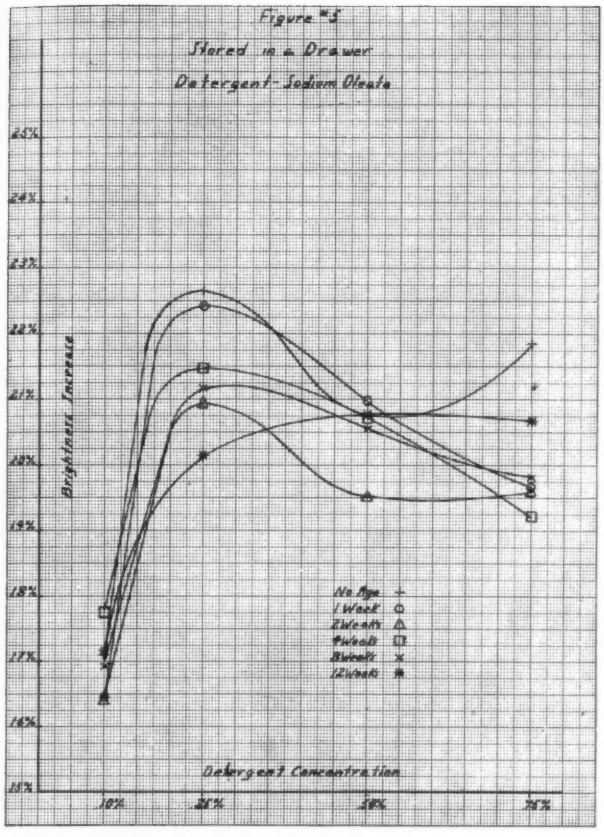



Figure #7

The Effect of Sample Area on the Removal of a Standard Soil

Detergent-Sodium Oleate

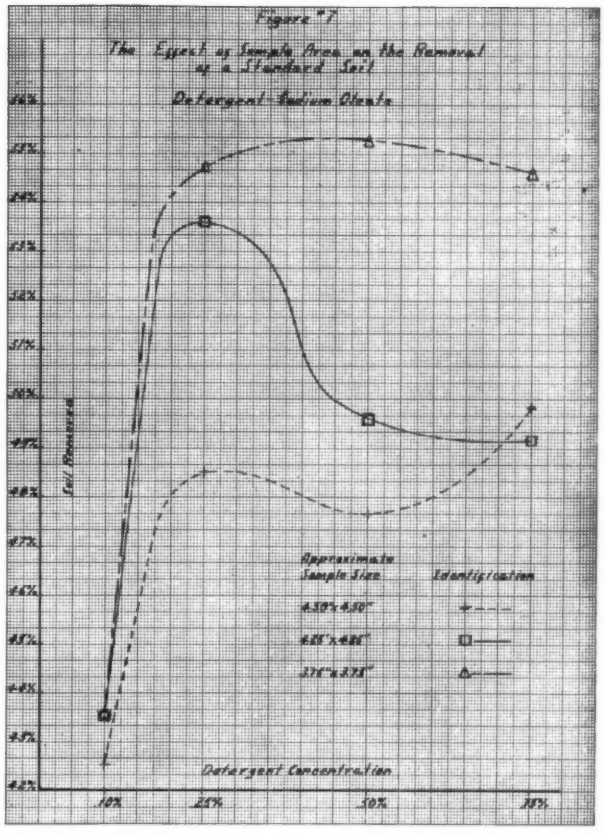



Figure #6

Stored in a Drawer

Detergent-Sodium Oleate

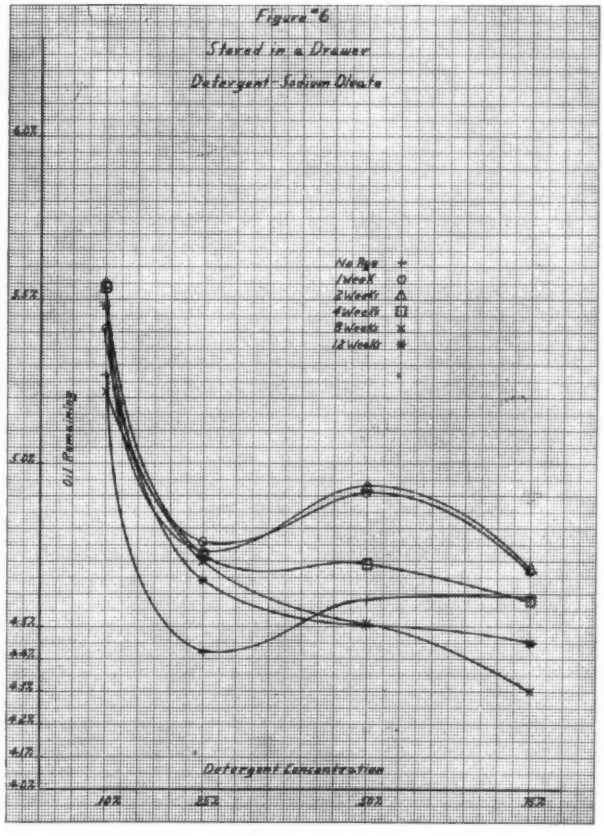
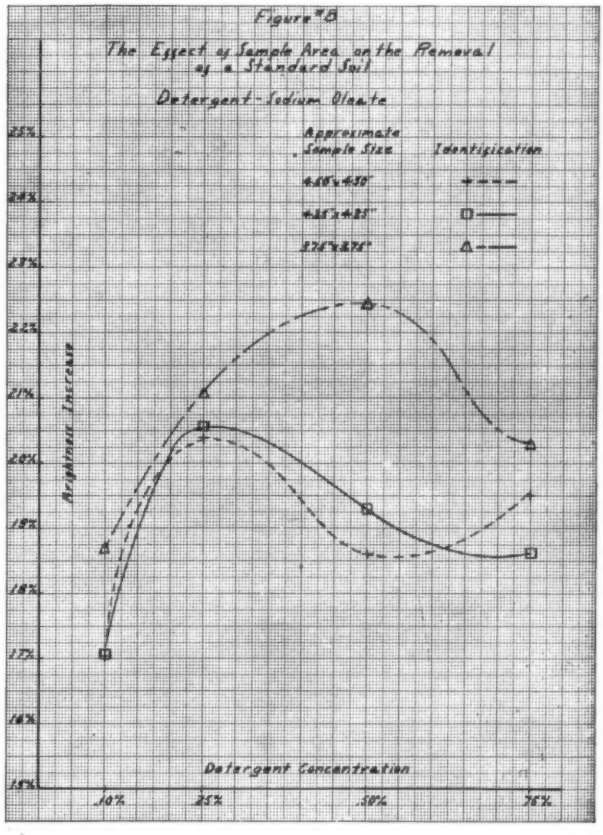



Figure #8

The Effect of Sample Area on the Removal of a Standard Soil

Detergent-Sodium Oleate



brightness. Soil removed is reasonably consistent also. The oil remaining calculations are apparently not quite so accurate. It is suggested that, since the oil remaining is generally the approximate complement to the soil removed, this evaluation factor be eliminated as superfluous. The fact that about 2/5 of the total evaluation time is taken up with this factor should be considered also.

Figure 2 compares favorably with the findings of Bacon (*1*) whereas figure 5 does not. This merely bears out the generally accepted fact that some soils are more stable than others. In the case of the soil used in this experiment, the writers believe that storage in an unsealed drawer would be sufficient for most detergent evaluations. This is based, in part, on the results shown in Table II. If a soil was made up of constituents possessing unsaturated bonds or oils that tended to volatilize, it is agreed that storage in a desiccator would be necessary.

## V. The Effect of Sample Area on the Removal of a Standard Soil

The object of this experimentation is to determine the effects in detergency evaluations of varying the size of the fabric samples, other conditions being constant. Logically, the larger the sample area, and the greater the amount of soil present as a function of that area, the less the soil removal becomes, the per cent brightness decreases proportionately, and the greater is the per cent of oil remaining.

*PROCEDURE:*

Samples of 64 x 64 sheeting 4.5 x 4.5, 4.25 x 4.25, and 3.75 x 3.75 inches were prepared, soiled, washed, and evaluated according to the method described under Part IV-B in the Procedure. The results of this work are depicted in Figures 7, 8, and 9.

*DISCUSSION OF RESULTS:*

The hypothesis advanced above appears to be in agreement with the experimental results, Percentage differences for the various sample sizes are in no case overwhelming, but are nonetheless present. Apparently, sample sizes are not too critical, but individual samples should always come as close to a standard area as possible for best results. Great differences of sample area cannot be tolerated, as is proven by the figures for the 3.75 x 3.75 inch samples as compared to the other two series, which are somewhat closer to each other in size and results.

## VI. Influence of Length of Wash on Soil Removal

Samples of fabric were prepared, soiled, washed and evaluated according to the method described under Part IV-B in the Procedure with the exception that the length of the wash period was varied.

The results of this experiment are shown in Table 3.

TABLE III

Effect of Length of Wash on Soil Removal
Detergent .25% Sodium Oleate

| Wash Time | % Soil Added | % Soil Removed | % Brightness Increase | % Oil Remaining |
|---|---|---|---|---|
| 15 Minutes .. | 8.02 | 46.90 | 20.29 | 4.60 |
| 30 Minutes .. | 8.80 | 50.30 | 21.38 | 4.74 |
| 45 Minntes .. | 8.78 | 51.51 | 20.59 | 4.57 |
| 60 Minutes .. | 8.25 | 50.65 | 20.78 | 4.55 |

It is generally conceded, all other conditions being constant, that a saturation or equilibrium point is reached after the lapse of a certain time period. Under the conditions of this part of the investigation, it is indicated that the equilibrium is reached in the vicinity of 30 minutes as regards soil removal whereas with the brightness increase of oil remaining, the peak seems to have been reached somewhere under fifteen minutes.

## VII. Relation of Temperature to Soil Removal

In this phase of the study cotton fabric was treated for detergency evaluation in the prescribed manner. (Procedure, Part IV-B) and divided into two sets. One set was laundered at a temperature of 125° F. and the other at 160° F. The results of these evaluations are portrayed in Table #4.

The general trend of thought is that increasing temperatures will yield greater soil removal. Contrary to this, the figures in Table #4 indicate that the reverse might be true. In order to verify these findings, the experiments were repeated and the results were found to check with the previous tests.

These unexpected findings are in all probability due to the physical characteristics of the detergent employed. Although surface tension and subsequent contact angles are greatly reduced at elevated temperatures, it may be that film strength and the carrying power of the detergent are impaired to the extent that the advantages of increased temperatures are overcome. The data above shows the necessity of evaluating a particular detergent at the temperature at which it is to be used in plant practice. Further, it suggests a means of determining at what temperature a detergent is most efficient.

## VIII. Affinity of the Standard Soil for Cotton, Wool and Synthetic Fibers

Until this point, all of the investigation has dealt entirely with soil on cotton fabrics. Due to their increasing importance to the textile industry, we shall now consider the possibilities of the Standard Soil for detergency evaluation on synthetic fibers as well as fiber blends.

*PROCEDURE*

Samples of the fabrics in question were scoured, desized, soiled and washed according to the methods as described under Part IV-B. The following are part of a list of blended fiber fabrics that were tested:

#1. Warp—cotton
Filling—50% Viscose
50% Cotton

#2. Warp—cotton
Filling—Viscose

#3. Warp—Cotton
Filling—50% Nylon
50% Cotton

#4. Warp—Cotton
Filling—50% Nylon
50% Viscose

#5. Warp—Cotton
Filling—75% Nylon
25% Viscose

#6. Warp—25% Wool
25% Nylon
50% Viscose
Filling—20% Wool
20% Nylon
60% Viscose

The results of this work are shown in figures 10, 11, and 12.

In figures 13, 14, and 15 are demonstrated the wash characteristics of pure nylon filament cloth as compared with fabric composed of nylon monofilament warp and spun viscose filling.

TABLE IV

Effect of Temperature on Washing Characteristics
Detergent—Sodium Oleate

| Temp. Degrees F. | Detergent Conc. | % Soil Added | % Soil Removed | % Brightness Increase | % Oil Remaining |
|---|---|---|---|---|---|
| 125 ..... | .10% | 8.72 | 48.08 | 15.78 | 4.91 |
| 125 ..... | .25% | 9.50 | 49.87 | 18.53 | 5.09 |
| 125 ..... | .50% | 9.29 | 46.32 | 16.28 | 5.25 |
| 125 ..... | .75% | 9.74 | 46.16 | 14.11 | 5.59 |
| 160 ..... | .10% | 9.97 | 42.53 | 13.92 | 6.21 |
| 160 ..... | .25% | 0.73 | 51.76 | 17.74 | 5.33 |
| 160 ..... | .50% | 9.64 | 45.97 | 16.66 | 5.50 |
| 160 ..... | .75% | 9.31 | 46.92 | 15.98 | 5.21 |

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| % Oil Remaining |
|---|
| 4.91 |
| 5.09 |
| 5.25 |
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| 5.50 |
| 5.21 |

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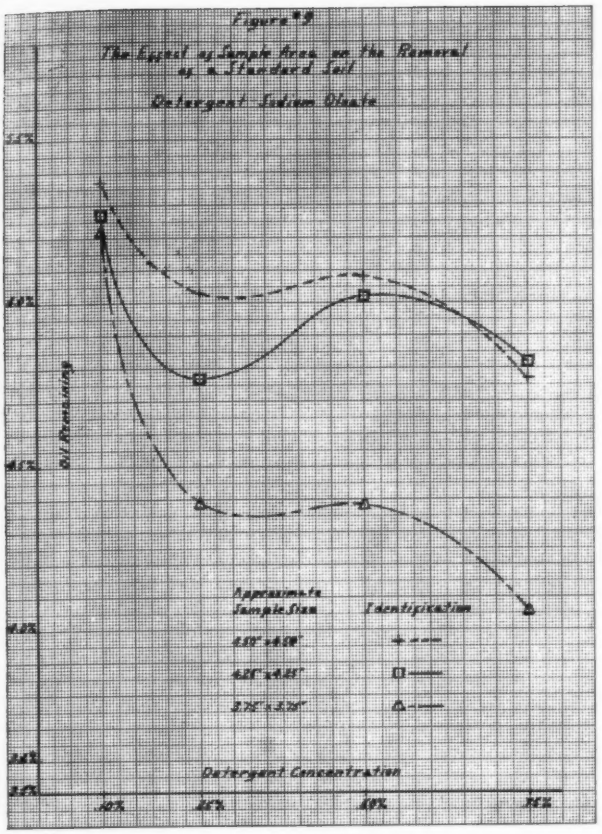


Figure "9
The Effect of Sample Area on the Removal of a Standard Soil
Detergent Sodium Oleate
Oil Removing
Approximate Sample Size
Identification
Detergent Concentration

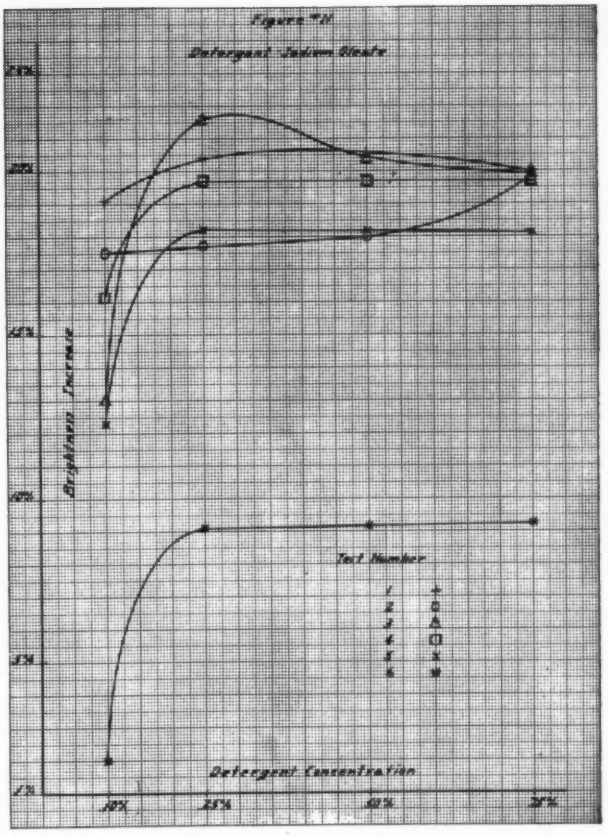


Figure "11
Detergent Sodium Oleate
Graphical Increase
Test Number
Detergent Concentration

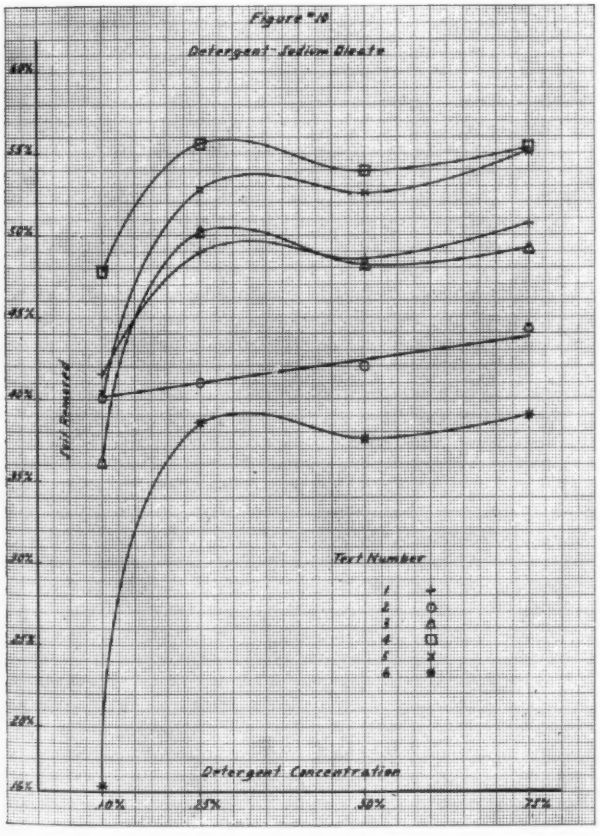


Figure "10
Detergent Sodium Oleate
Oil Removed
Test Number
Detergent Concentration

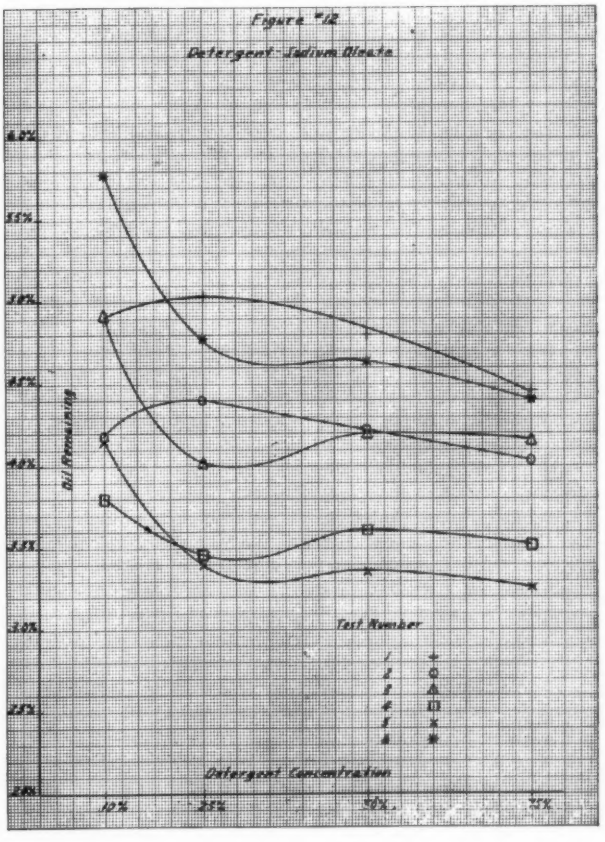


Figure "12
Detergent Sodium Oleate
Oil Removed
Test Number
Detergent Concentration

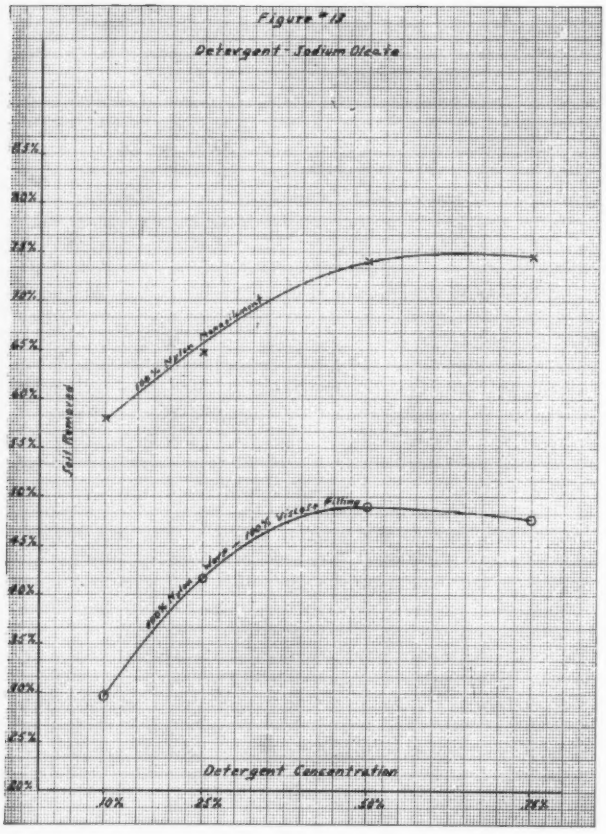


Figure #13
Detergent - Sodium Oleate
Soil Removed
100% Nylon Monofilament
100% Nylon Yarn — 100% Viscose Filling
Detergent Concentration
10%
25%
50%
75%

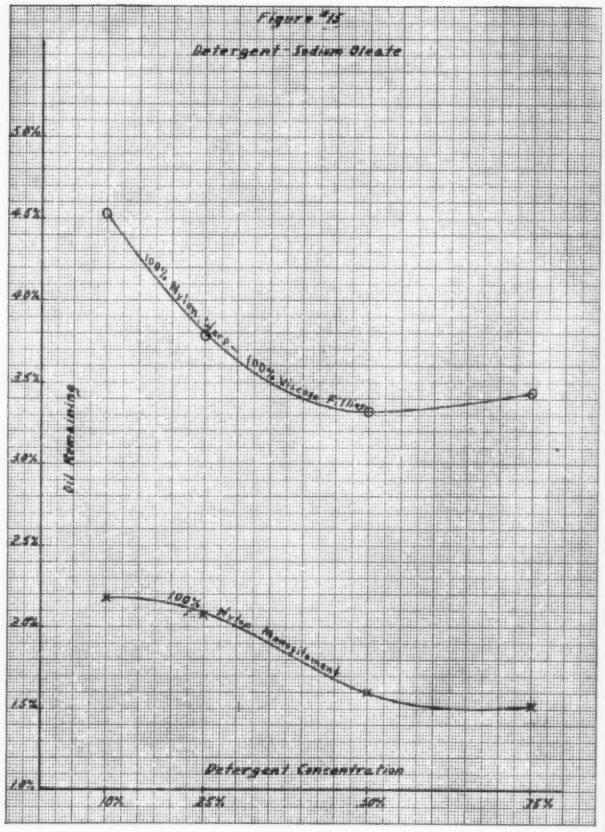


Figure #15
Detergent - Sodium Oleate
Soil Remaining
100% Nylon Yarn — 100% Viscose Filling
100% Nylon Monofilament
Detergent Concentration
10%
25%
50%
75%

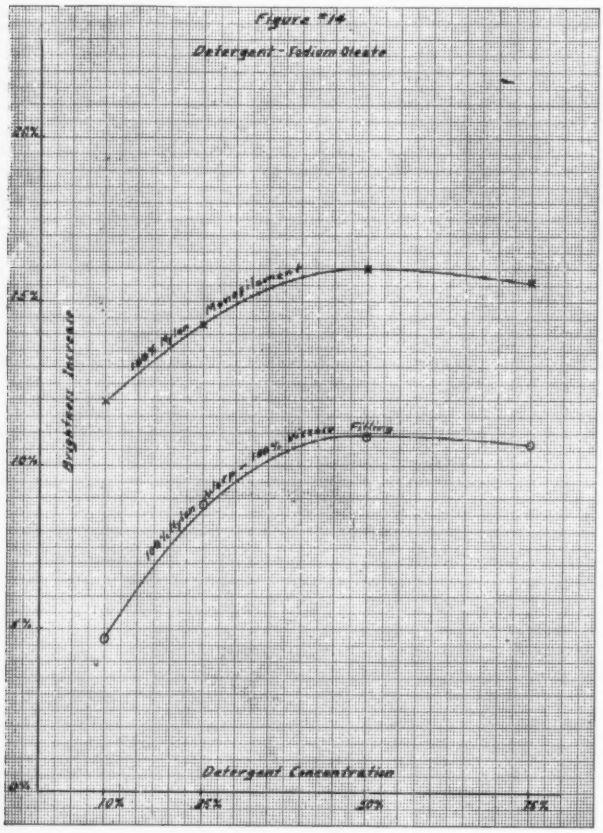


Figure #14
Detergent - Sodium Oleate
Brightness Increase
100% Nylon Monofilament
100% Nylon Yarn — 100% Viscose Filling
Detergent Concentration
10%
25%
50%
75%

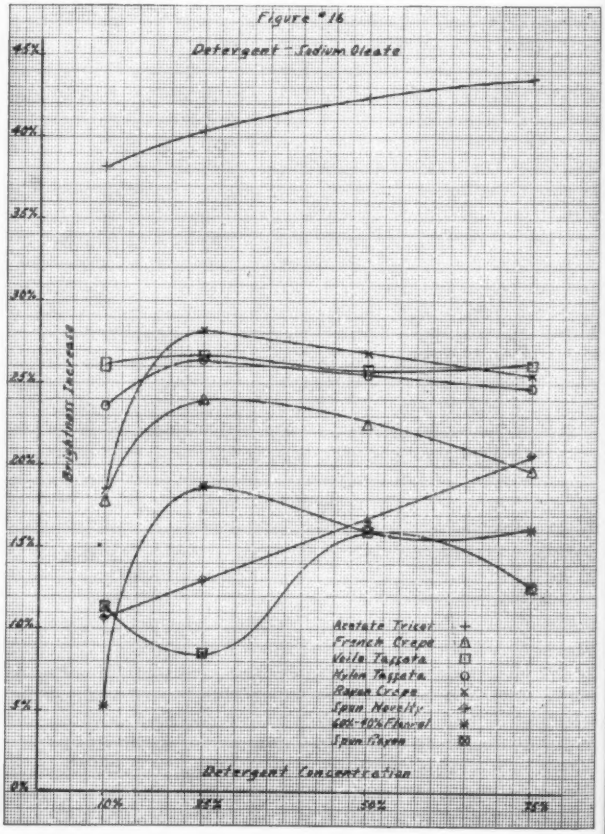
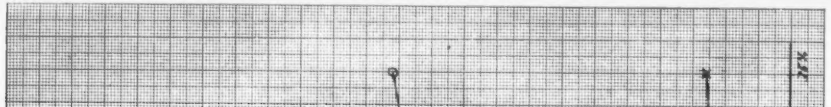
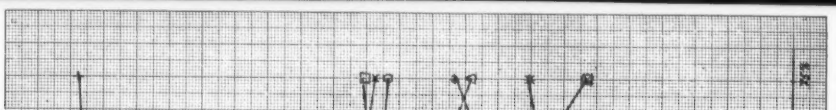


Figure #16
Detergent - Sodium Oleate
Brightness Increase
Acetate Tricot
French Crepe
Voile Taffeta
Nylon Taffeta
Rayon Crepe
Spun Novelty
60%-40% Flannel
Spun Rayon
Detergent Concentration
10%
25%
50%
75%





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Figures 16, and 17 illustrate the wash-characteristics of the standard soil on various fabrics of diversified construction and fiber composition.

*DISCUSSION:*

The results shown in Figs. 10, 11, and 12 indicate that the removal of the Standard Soil, under the conditions of the test, from these fabrics composed of fiber blends are, with one exception, fairly well in line with the results obtained with an all cotton fabrics. The exception to the trend in this case is that blend containing wool. This deviation can be attributed in part to the fact that the yarn in this sample of fabric was appreciably heavier than the yarn in the other fabrics of the series. Further, the nature of the wool fiber does not lend itself to reflectivity as well as the other fibers.

The tests involving the monofilament nylon as brought out in Figs. 13, 14, and 15 reveal some interesting results. Exceptionally high removal of the Standard Soil is experienced with the 100% nylon monofilament sample whereas the fabric containing the same nylon as the warp and spun viscose as the filling is not any better than the results obtained with cotton under the same conditions. The nylon samples also lend themselves to easier oil removal and a better brightness increase than do the nylon-viscose samples. Although the oil remaining in the all nylon samples are considerably lower, their brightness increase is much lower than cotton. In general, this holds true for the fabrics containing blends with cotton as described previously.

In conducting the tests with the monofilament series as above, it was noted that the characteristics of the fabric weave were such that it was possible to get a high and low reflectometer reading that would check on both sides of the particular sample. It was found that care must be taken that all readings on a series should be taken with the samples placed in the reflectometer in exactly correspoding positions according to warp and filling.

In the results shown in Figs. 16 and 17 none of the samples, with the exception of the *all acetate tricot*, demonstrated any unusual phenomena. The tricot gave exceptionally high soil removal values, probably because of its extreme limpness. Obviously the weave and not the fiber is the cause of this since the *acetate voile taffeta* gave very average results. With this exception, any of the fabrics listed in these two graphs would seem suitable as a basis of detergency evaluation under the conditions of the described procedure.

## IX. Evaluation of Four Synthetic Detergents

To illustrate the use of the standard soil and procedure developed in evaluating products other than soap, four synthetic detergents were selected and tested according to the method already described. The characteristics of these materials are compared in Figs. 18, 19 and 20.

These curves are particularly interesting in showing the two extremes of detergent efficiency. Out of a considerable number of synthetics evaluated, Synthetic #3 was outstandingly the best and Synthetic #4, the poorest. In the case of soil removal, the results from Synthetic #4 ranged from 26 to 30% and could not be conveniently shown on the graph. Observe how the curves of Synthetic #3 level off sharply at the 0.5% concentration denoting the point of maximum efficiency.

## X. Discussion and Summary

In the evaluation of any detergent the job to be done must be given first consideration. With this in mind, the soil employed should approach the soil to be encountered in plant processing as closely as possible as long as these conditions do not effect soil stability and accuracy of method adversely beyond a limited degree. The foregoing experiments have shown that the type of soil, sample area, length of wash, washing temperature and type of fabric do have a pronounced effect on removal of soil from fabric by any detergent. Therefore the textile chemist should apply the temperature, time, water conditions, fabric, etc. a detergent is expected to meet in the mill in his method of evaluation.

The application of any standard soil to fabrics for the purpose of detergency evaluation does require the development of technique and a certain amount of skill if a fair degree of accuracy is to be maintained. A goodly number of textile concerns do have small wash wheels or launderometers, but no facilities for the proper treatment of fabric with so-called standard soils. For this reason it is suggested that the AATCC make available to its membership, as well as interested parties, standard soiled fabric for the purpose of detergent evaluation. This could be extended from a single soil on one type of fabric, to a single soil on several types of fabrics or a series of soils on a series of fabrics. In addition, it may be advisable to offer a service whereby the reflectivity of the samples, before and after laundering is measured by the research department of the association for a nominal fee.

During the course of this study where a large number of tests were run in the Launder-Ometer, two difficulties were encountered which should be mentioned. One of these was belt slippage. After long periods of running under full loads, imperceptible slippage occurred with the consequence that the speed was slower with a full complement of jars and increased as the jar load lightened. This produced unaccountable results until the trouble was found. For accuracy put on a new belt and be sure that it does not slip.

The other difficulty was found in temperature control. This was not due to any inherent weakness in the launderometer, but rather to the wear and hard usage to which the control had been subjected. Temperature should be kept under close observation. To establish accurate temperature control the regular launderometer control was disconnected and replaced with a Brown Automatic Temperature Controller and Recorder which was used in all the preceding evaluations.

In the foregoing work some of the factors involved in detergency have been considered. The results of these experiments leaves no doubt that there is much to be done before a satisfactory understanding of the term "detergency" can be attained. In the dictionary we find that a detergent is a material having cleansing qualities. The question in hand is what are cleansing qualities, what are their functions, and how may they be measured?

Whenever detergency is discussed, it is usually in terms of factors affecting detergency. Snell (*8*) states the factors affecting detergency are (1) pH of the detergent solution, (2) buffer value (3) interfacial tension and (4) emulsifying power. He believes that if one detergent raises the brightness of a soiled fabric from 30 to 40 and another from 30 to 50, rather than being twice as good, the second is not a linear function, but is logarithmic and is something like four times as good.

Bacon and Smith (*2*) have offered a new working hypothesis in explaining detergency. They bring out that "the effectiveness of surface-active agents in detergent action is related quantitatively to the amount of work required to remove a fixed amount of a standard soil from a cotton fabric. The factors of detergent concentration, relative mechanical force of the washing machine, and time of washing are inversely proportional to each other at the same degree of soil removal." In the tests of this paper the work done by the washing machine has been constant, the amount of soil on the fabric constant, while the detergent concentration varied. This, in effect, parallels the report of Bacon and Smith.

In this study an attempt has been made to touch upon some of the outstanding factors that influence the evaluation of de-

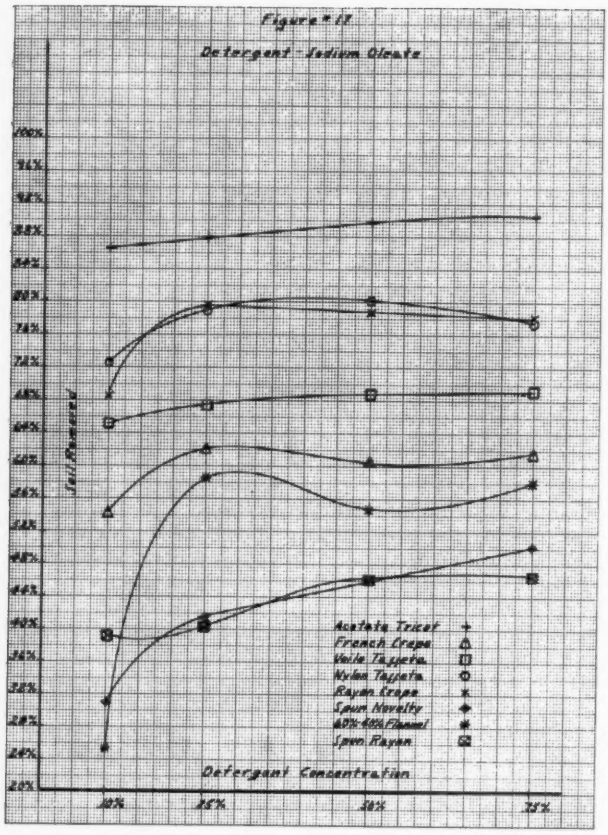
Figure "17
Detergent - Sodium Oleate
Soil Removed
Detergent Concentration
Acetate Tricot
French Crepe
Voile Taffeta
Nylon Taffeta
Rayon Crepe
Spun Novelty
60% 40% Flannel
Spun Rayon

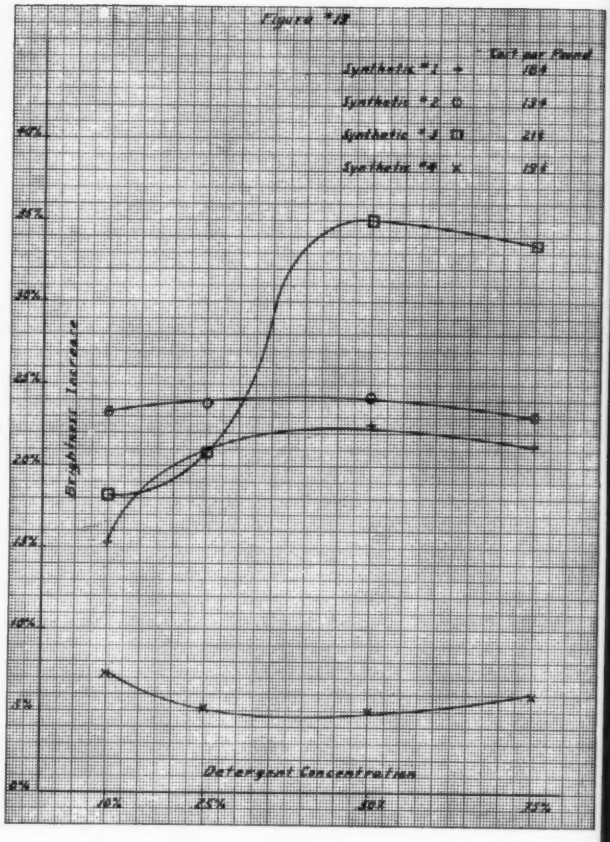
Figure "18
Synthetic "1 +
Synthetic "2 ○
Synthetic "3 □
Synthetic "4 ×
Brightness Increase
Detergent Concentration

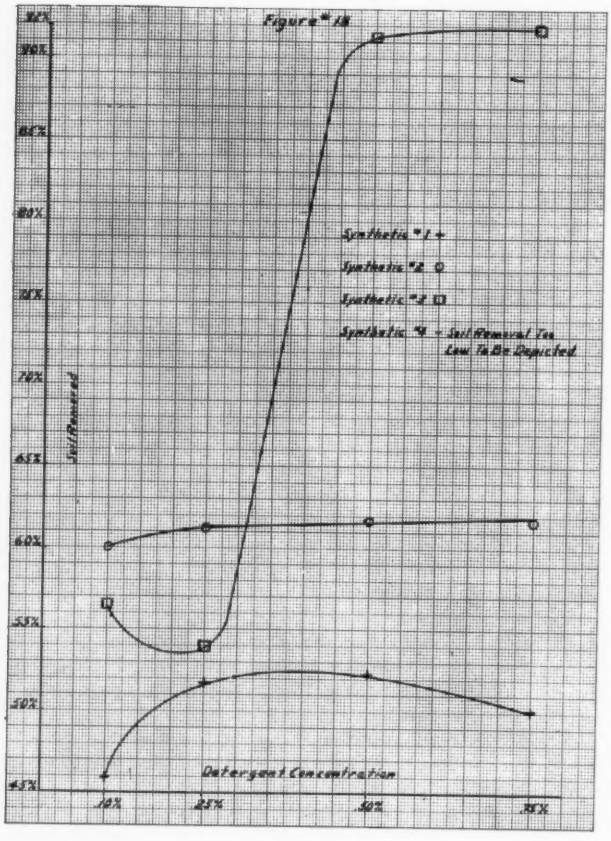
Figure "19
Synthetic "1 +
Synthetic "2 ○
Synthetic "3 □
Synthetic "4 = Soil Removal Too Low To Be Depicted
Soil Removed
Detergent Concentration

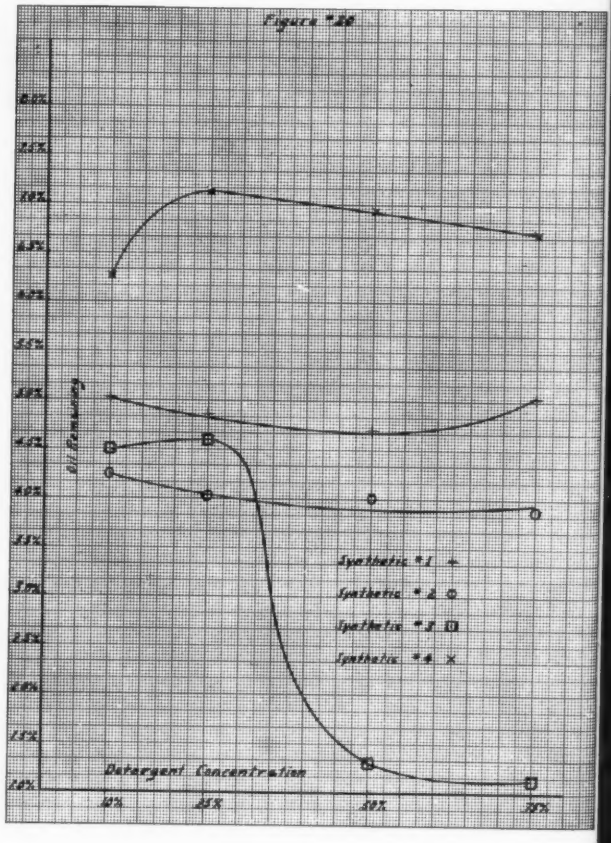
Figure "20
Oil Removed
Synthetic "1 +
Synthetic "2 ○
Synthetic "3 □
Synthetic "4 ×
Detergent Concentration

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tergents for textile processing. Many questions are left unanswered and it remains for the development of improved test methods, specialized instruments, and considerable concerted effort on the part of research groups to bring to light a more complete understanding of the problem. In any case, a tentative standard procedure for evaluating detergents should be set-up so that some working basis can be realized.

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(10) Vaughn, Thomas H., Vittone, Auton Jr., and Bacon, Leslie R.: Ind. & Eng. Chem., Vol. 33, No. 8, Aug., 1941.
(11) Woodhead, J. A., Vitale, P. T., and Frantz, A. J.: Oil and Soap 21, 3337 (1044).

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# GENERAL TECHNICAL SESSION

*National Convention, October 24*

THE General Technical Session convened at nine-forty o'clock in the Congress Hotel, Chicago, Illinois, Dr. Miles A. Dahlen, National Chairman, Technical Program Committee, presiding.

*Chairman Dahlen:* The meeting will come to order.

First of all, I can assure you it is a pleasant surprise to all of us to see so many of our members at this meeting out here in the West. Having had a part in preparing the Technical Program, I should like to think that many of you have been attracted by the technical side of the meeting just as much as you have been attracted by the famed hospitality of Chicago.

While I do not wish to consume any of the time reserved for our speakers, I do wish to take a few minutes to explain briefly how this Technical Program was prepared. I am doing this because we were confronted by many questions and had to make arbitrary decisions which may please some of you and displease others.

Your President appointed me National Technical Program Chairman and provided me with an excellent committee, having representation from each Section of our Association. Here in Chicago, Herman Boxser is serving as Program Chairman for this Convention, and he also is the Chicago member of the National Committee.

Practically our entire committee met in New York late last spring to make general plans. We had to choose between (1) two general technical sessions at which a few papers of general interest could be presented, or (2) break up our membership into small groups according to their specific interests. We decided to compromise. This morning's meeting is composed of three outstanding papers of general interest. This afternoon, we shall separate into six group meetings, making the best division among the major interests of our members which we could devise. We know that some of you will object to these group meetings. Certain individuals will be disappointed because they cannot hear papers in different groups at the same time. Our only answer is that without this arrangement, some of the papers would have to be omitted entirely. At worst, you will be able to read the papers in the AMERICAN DYESTUFF REPORTER, if you cannot have the pleasure of attending.

I might add at this point that the group papers will be spaced approximately forty-five minutes apart; hence, if you wish to move from one group to another, you can make a reasonably accurate calculation as to when the papers you wish to hear will be presented.

It is our hope that the group meetings will provide active discussion and that there will be a free exchange of ideas and information on the many subjects being covered.

Another criticism we expect to hear of this program is that too many of the papers are being presented by suppliers of chemicals and dyes and too few by men in the dyeing and finishing industry. It was our hope that we could have many speakers from the latter group. It was with this idea in mind that two full-page announcements appeared in the AMERICAN DYESTUFF REPORTER last summer, outlining the nature of this program and inviting contributed papers.

I regret to report that as a result of these announcements, we received not a single contributed paper from the mills. In fact, these announcements have served well as a reference to the nature of the meeting and the principles involved in presenting papers; but as far as bringing in papers are concerned, they were practically a failure. Exactly two of the papers on the entire program were obtained in this way. All of the remaining ones were obtained by the active solicitation of the program committee members. At this time I want to express my sincere thanks to all of this committee, and especially to Mr. Boxser in Chicago, for the excellent work in developing this program.

The chairmen of the group meetings this afternoon are all Midwesterners and a very capable group. I also wish to thank them for their good services, and particularly do I wish to thank our many speakers who, although most of them did not volunteer to give papers, willingly and gladly accepted our invitations and I am sure are bringing us much valuable information.

I should like to ask that all of you give considerable thought to this question of Technical Programs at National Meetings, and that you express your opinions and suggestions during the group meetings this afternoon. I am asking the group chairmen to solicit such comments and to summarize your ideas for the guidance of the next program committee. We sincerely hope that at the next Convention this committee will be able to present a program with a larger proportion of papers by members of the dyeing and finishing industry.

Without further preliminaries, we shall now proceed with our program for this morning. Three papers will be presented, which are believed to be of general interest and are very different in their subject matter.

The first paper is intended to bring to your attention an exceedingly important new chemical development. Our technical work in the past has been concerned largely with the use of organic chemical compounds, meaning compounds of carbon. However, we all learned in school from the "Periodic Table of the Elements" that there is another chemical element, silicon, directly under carbon, with analogous properties. Within the past few years, the new organic chemistry of silicon has been developed in our laboratories and it is growing rapidly. The compounds of silicon are certain to make an important contribution to textile processing.

Our first speaker is F. L. Dennett of the Dow Corning Corporation, Midland, Michigan.

Mr. Dennett is a graduate in Pulp and Paper Technology from the University of Maine, and has a Master's degree in Pulp and Paper Manufacture from the New York State College of Forestry.

Upon receiving his degree, he was employed by The Dow Chemical Company; and for four years he was in their Cellulose and Plastics Division. In 1942, he began work on the silicones and in 1944 joined the Dow Corning Corporation.

He has had wide experience in the production of silicon compounds and their application. I am sure that Mr. Dennett is bringing us a very important story in his paper entitled, "Silicones and Their Application to the Textile Industry."

## General Technical Session—

# Silicones

## and their Application to the Textile Industry*

F. L. DENNETT

*Dow Corning Corporation, Midland, Michigan*

### Introduction

THE spinning of yarns and the weaving of fabrics were among the first of the arts. The industrialization of these arts marked the beginning of the Industrial Revolution. Through all of its long history, the textile industry has recognized the value of scientific and technical knowledge. This is evidenced by your continued search for textile improvement and by your cooperation with the chemist in the development of synthetic fibers and plastic finishes.

There is now a new family of chemical materials which promises to be of great importance to the textile chemist and to the operators of textile mills. These materials are called silicones[1] and are different from any material found in nature or created synthetically. They are different because they are semi-inorganic in chemical structure. Their properties therefore lie midway between those of such inorganic materials as quartz and glass and the many organic plastics.

This paper will describe the basic properties of these new materials, giving particular emphasis to those applications which are significant to the textile industry.

### History of Silicones

The term 'silicone' was first used by Prof. F. S. Kipping[2] of the University of Nottingham, England to describe some of the lower polymeric materials which he obtained upon the hydrolysis of disubstituted silicon dichloride. We now use this term to describe the entire group of organosilicon oxide high polymers.

During the period of 1899 to 1935, Prof. Kipping gathered a great quantity of information about organo-silicon compounds which served as a foundation for the development of the complex and useful silicone molecules described in this paper. In the early 1930's research in the field of organo-silicon compounds was undertaken at the Corning Glass Works and at the Mellon Institute of Industrial Research to develop materials useful as coatings for glass fabrics and as adhesives for glass blocks. As this work progressed a very essential group of engineering materials emerged. At present there are four quite distinct classes of silicone products: fluids; greases and compounds; resins and varnishes; and the silicone rubbers.

### Silicone Chemistry

The first step in the manufacture of silicones is the conversion of silicon dioxide to silicon tetrachloride through the use of coke and chlorine as shown in Fig. 1. The next step is to replace one or more of the chlorine atoms of the silicon tetrachloride with organic radicals. This is accomplished by reacting chlorinated hydrocarbons with magnesium to form the familiar Grignard reagent, and in turn reacting this with silicon tetrachloride[3]. This complex chemistry yields the organosilicon chlorides.

These chlorosilanes are intermediates in the production of silicones and their properties are entirely different from those of the end product. The chlorosilanes are volatile, toxic, and acid. They have been used however as a vapor treatment[4] to make glass and ceramic insulators water repellent. These vapors hydrolyze on the surface to form a hydrophobic silicone

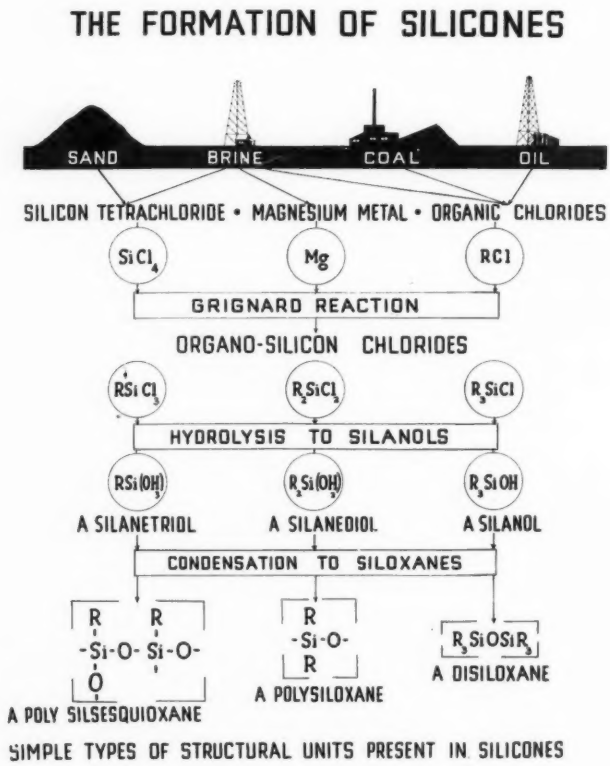


*Figure 1*

* Presented at General Technical Session, National Convention, Chicago, October 24, 1947.

polymer and hydrochloric acid. Some efforts have also been made to use the chlorosilanes as a water repellent treatment for paper and textiles. This material is unsuited to such applications, however, because the hydrochloric acid may damage the fibers in spite of all efforts to neutralize the acid.

In the production of silicones, these chlorosilanes are first hydrolyzed to remove the chlorine as hydrochloric acid. A condensation reaction follows which builds molecular structures of alternate silicon and oxygen atoms having one or more hydrocarbon radicals bonded to the silicon[5, 6]. The length of the polymer chain, the degree of cross linking, and the kind and number of hydrocarbon groups attached to the silicon atom determine the properties of the finished polymer.

## Silicone Resins and Varnishes

One of the first problems assigned to the research group under the direction of Dr. J. F. Hyde at Corning Glass Works was the development of a silicone resin having sufficient heat stability to seal the voids in the glass fabrics designed for use as insulating materials in electric motors, generators, and transformers.

This research group developed the first useful, cross-linked organosilicon oxide polymers, and presented these materials to the electrical industry for evaluation. These resins were so promising that Dow Corning Corporation was formed in 1943 to produce silicones in commercial quantities. To accomplish that purpose Dow Corning built the first plant designed exclusively for the production of silicones and had it in operation by the Fall of 1944.

Glass cloth together with asbestos and mica gave the electrical industry all of the heat stability they needed in insulating materials. But the permissible operating temperatures were still limited by the relatively poor heat stability of the organic resins and varnishes used to bond and impregnate the glass, mica and asbestos. The new silicone insulating resins and varnishes[7, 8] proved to be natural complements to such inorganic insulating materials. Because they have the same skeletal structure of silicon and oxygen atoms found in these inorganic materials, silicone resins are resistant to temperatures 200° F. above the limits of organic resins which are based on the less stable carbon to carbon linkage.

Tests indicate that silicone resins and varnishes used to bond these inorganic insulating materials may withstand as much heat as the metals used in building electrical machines. In our laboratories we operated a group of test motors at 600° F. for 18 months. The silicone insulation was still intact but the motors failed due to oxidation of the copper at these high temperatures. As a result of the comparative testing[9] which we have done during the past five years, electrical engineers state that Silicone Insulation has at least ten times the life and ten times the wet insulation resistance of the best class of electrical insulation previously known. For this reason increasing use is being made of Silicone Insulation in rewinding old motors and in building new motors and other electrical equipment. Standards[10] for Silicone (Class 'H') Insulation have recently been set by the American Institute of Electrical Engineers.

Silicone Insulation is well suited to the severe operating conditions of motors driving textile machinery. The fire resistance of a silicone-glass insulated motor is also a very important point to be considered. We feel that silicone insulation can help you in every case where you have insulation failure in motors due to overloads, high surrounding temperatures, and humid, lint filled, and corrosive atmospheres.

For example, an interesting application we have found for Silicone Insulation is the protection of spinning bucket motors in synthetic rayon plants. These motors are subjected to operating conditions so severe that in one plant alone over a thousand of them have had to be rewound every month. Corrosive chemicals and high humidities penetrate the windings. They have a high starting load and when they stall, the temperature in the windings goes up as high as 1100°F. burning out the conventional insulation in five minutes. One Silicone Insulated motor, tested under the above conditions, has shown 48 times the heat resistance of the conventional motors.

In addition to the silicone insulating varnishes and resins there are thermosetting silicone resins[11] used for bonding inorganic materials in the fabrication of rigid laminations. These laminates are designed primarily for electrical applications. Another series of silicone resins[12] is used in formulating heat and weather resistant protective coatings. Properly applied and cured, they will withstand temperatures ranging up to 500°F. As vehicles for aluminum powder, temperatures up to 1000°F. are within their range. They endure outdoor weathering without chalking or yellowing, and have good resistance to a wide variety of chemical fumes. They are being used as protective coatings on ovens, boilers, smoke stacks and heating ducts. They should find many uses in textile mills as protective coatings for curing ovens and other equipment operating at high temperature and exposed to chemical fumes or high humidities.

## Silicone Greases and Compounds

The development of silicone insulation, which permitted motors to be operated at higher temperatures, made it necessary to develop a grease able to withstand the higher operating temperatures of the motor bearings. As a result we have compounded a group of greases[13] from the silicone fluids. These greases have found a variety of industrial uses, in addition to solving the problem of lubricating anti-friction bearings operating at temperatures as high as 400°F. Comparative tests indicate that these greases have about ten times the life of special high temperature petroleum products. They are now being used in sealed ball bearings because they appear to be the most nearly permanent lubricants available. You men who are confronted with lubrication problems involving high temperature and high humidity as in tenter frames, conveyors, and dryers may find your answer in these new greases.

One of the most interesting and valuable of the silicone compounds is a defoaming agent[15] which has proved to be very effective in a wide variety of aqueous systems even during steam distillation or vacuum evaporation. It is used to kill foam in the processing of synthetic rubber, paper, paint and varnish, asphalt and adhesives. This silicone compound should be similarly useful in killing foams in textile sizings and finishes, printing pastes, adhesives and coatings. It is generally effective in dilutions ranging from 1 part in 10,000 to 1 part in 1,000,000.

## Silicone Rubber

Elastic silicone materials, commonly called silicone rubber,[14] are produced by carrying out a more orderly and limited cross-linking than that required for silicone resins. These silicone elastomers are almost entirely inorganic in composition. Their basic silicon-oxygen-silicon structure enables them to retain their properties during continuous exposure to temperatures ranging from —70° F. to 300° F. They are water repellent, and weather resistant. Compounds recommended for fabricating gaskets have good resistance to a wide variety of chemicals. Other stocks are used as coatings for fabrics in the production of gaskets, diaphragms, conveyor belts and electrical insulating tapes. These elastic silicone materials can be molded, extruded, coated or used as calking and potting compounds.

Silicone elastomers, however, do not have the mechanical strength of organic rubbers at normal operating temperatures.

They are not recommended, therefore, as a general substitute for natural or synthetic rubber, but their good electrical properties, weather resistance and stability to temperatures far above and below the limits of organic rubbers recommend them for a wide variety of industrial uses.

## Liquid Silicones

Long chain silicone polymers are produced as water white fluids[17] in a wide range of viscosities depending upon chain length, and in several different types depending upon the kind of organic radicals attached to the silicon atoms. In general, the silicone fluids are much less volatile, have higher flash points, lower freezing point and greater resistance to oxidation than petroleum oils of equal viscosity. They are water repellent and electrically non-conductive. They have a more nearly constant viscosity over a wider temperature range than any other fluid. The viscosity-temperature slopes of some of these silicone fluids are compared with various petroleum oils in Figure 2.

The properties of these various silicone fluids are given in Table I. They are used as damping and hydraulic fluids, liquid dielectrics, mold release agents, antifoaming agent for petroleum oils, lubricating oils, diffusion pump fluids, lens cleaners, and water repellent treatments for glass and ceramic insulator bodies.

Among the many industrial uses for these various silicone fluids, only three are of special interest to textile chemists.

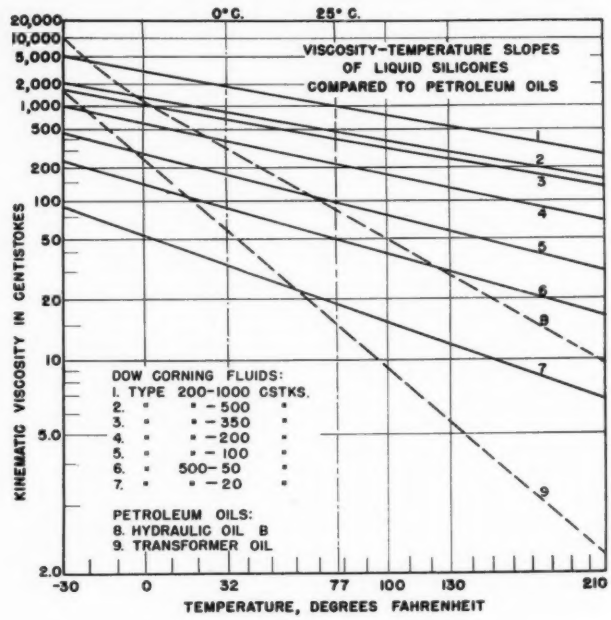


*Figure 2*

First, the fact that silicone fluids are widely used as mold release agents for metal dies used in molding synthetic rubber and organic plastics, suggests that this lubricity toward plastic materials might be utilized for lubricating such thermoplastic fibers as Saran, nylon, Vinyon and acetate rayon.

Second, some of the silicone fluids, notably DC 550 and DC 710 have good lubricity under light to moderate loads and between certain combinations of metals. DC 710 mixed with finely divided graphite is used as a lubricant for conveyor bearings and other machinery operating at extremely high temperatures. Comparative tests indicate that in roller bearings subjected alternately to temperatures of 350° F. and to 100% relative humidity, silicone oils have from 8 to 12 times the

TABLE I

Physical Properties of Dow Corning Silicone Fluids

| *Uses of Dow Corning Fluids* | *Fluid Type* | *Viscosity Grade* Cstks. at 25°C. | SSU at 100°F. | *Freezing Point* °C. | °F. | *Boiling Point* Temp. °C. | Pres. mm. Hg. | *Min. Flash Point* °F. | *Lbs. per Gal.* at 25°C. | *Refractive Index* 25°C. |
|---|---|---|---|---|---|---|---|---|---|---|
| Damping Fluids | | 0.65 | 29 | −68 | −90 | 99.5 | 760 | 30 | 6.35 | 1.3748 |
| Hydraulic Fluids | | 1.0 | 30 | −86 | −123 | 152 | 760 | 100 | 6.84 | 1.3822 |
| | | 1.5 | 31 | −76 | −105 | 192 | 760 | 160 | 7.11 | 1.3872 |
| Instrument Liquids | DC 500 | 2.0 | 32 | −84 | −119 | 230 | 760 | 195 | 7.27 | 1.3902 |
| | | 3.0 | 34 | −70 | −94 | 70–100 | 0.5 | 225 | 7.48 | 1.394 |
| | | 5.0 | 40 | −70 | −94 | 120–160 | 0.5 | 270 | 7.67 | 1.397 |
| Liquid Dielectrics | | 10 | 52 | −67 | −88 | >200 | 0.5 | 350 | 7.85 | 1.399 |
| Foam Inhibitors | | 20 | 78 | −60 | −76 | >200 | 0.5 | 520 | 7.93 | 1.400 |
| | | 50 | 198 | −55 | −67 | >250 | 0.5 | 540 | 7.93 | 1.402 |
| | | | | *ASTM Pour Point** | | *Volatility on Heating 48 Hrs.* | | | | |
| Mold Release Agents | | | | °C. | °F. | at °C. | % | | | |
| Waterproof Treatment for Glass and Ceramic Insulators | | 100 | 355 | −55 | −67 | 200 | <2 | 600 | 8.08 | 1.4030 |
| | | 200 | 715 | −53 | −63 | 200 | <2 | 615 | 8.10 | 1.4031 |
| Special Lubricants for Glass, Rubber and Plastics | DC 200† | 350 | 1250 | −50 | −58 | 200 | <2 | 625 | 8.11 | 1.4032 |
| | | 500 | 1750 | −50 | −58 | 200 | <2 | 625 | 8.11 | 1.4033 |
| | | 1000 | 3500 | −50 | −58 | 200 | <2 | 640 | 8.12 | 1.4035 |
| High Temperature Baths | DC 550 | 100–150 | 300–400 | −48 | −54 | 250 | <10 | 600 | 8.90 | 1.498 |
| General Lubrication at High Temperature | DC 710 | 500 | 1060 | −12 | 10 | 250 | <15 | 520 | 9.27 | 1.539 |
| | | *Viscosity in Cstks. at* °C. | 25°C. | 100°C. | *Freezing Point* °C. | °F. | *Boiling Point* °C. | °F. | | |
| Heat Transfer Media | DC 701 | 52 | 9.2 | 2.2 | −65 | −85 | 320 | 760 | 291 | 8.51 | 1.4800 |
| High Vacuum | DC 702 | 290 | 27.0 | 3.7 | −40 | −40 | 400 | 760 | 381 | 8.91 | 1.5062 |
| Diffusion Pump Oils | DC 703 | 950 | 60.0 | 6.3 | −36 | −33 | 430 | 760 | 441 | 9.05 | 1.5171 |

* DC 200 Fluids are not recommended for use below about −40°C. (−40°F.).
† DC 200 Fluids can be made available in higher viscosities to meet specific use requirements.

life of high temperature petroleum oils.

The third application and the one of greatest interest to textile chemists, is the use of various silicone fluids to impart water repellency to glass and ceramic surfaces, glass fabrics, and textiles.

## Treatment of Fibrous Materials with Silicones

All of the silicones are characterized by a very high order of water repellency. This property is probably due to the peculiar orientation of the hydrocarbon groups which are bonded to the silicon atoms. The degree of water repellency varies somewhat with the chemical structure of the various silicones and, in most cases, heat alters the orientation of the hydrocarbon radicals to increase the water repellency of the silicone treated surface. Many of the industrial uses which have been found for silicone products depend to some extent upon this hydrophobic property.

The first specific use made of this property was in the treatment of glass and ceramic insulator bodies[18]. In this application glass or ceramic insulators are dipped in a solvent solution of a silicone fluid and baked for half an hour at 300° C. to bond the silicone coating to the insulator surfaces and orient the hydrocarbon groups to give maximum hydrophobing. Such silicone treated surfaces have excellent water repellency as illustrated in Fig. 3. This silicone surface is not removed by neutral solvents or by detergents. It maintains high surface resistivity even after long immersion in sea water.

This early application to glass led to a study of the hydrophobic properties of several different silicone polymers[19]. The contact angle between water and clean glass slides treated with methylsilicone and stearyl silicone are plotted in Fig. 4. This chart shows that after heating, both of these materials produce contact angles greater than 90°. A contact angle of 108° is produced by methylsilicone after 15 minutes of heating at 300° C.

As a basis for comparison, the contact angle for high grade paraffin when applied and tested at room temperatures is 105°. Paraffins, which were considered to be among the most water repellent materials, prior to the introduction of silicones, have the obvious disadvantages of poor solvent resistance and no heat stability.

Another and more recently developed use of the hydrophobic properties of the silicones is in the treatment of glass fabrics. In this application a silicone oil emulsion is padded onto the glass fabric and cured for a short time at a high temperature. This treatment is remarkably resistant to solvents and detergents. It makes the glass fabric thoroughly water repellent and gives it a soft hand, and excellent draping qualities.

## Water Repellent Silicone Finish for Textiles

Since the glass fabric and the silicone polymers are both heat-stable, the maximum water-repellency of the methylsilicone polymer can be developed by exposure to high temperatures without damaging the fabric. Organic fibers of course could not withstand such temperatures. The problem then was to develop a silicone material which would make organic textiles water repellent without damaging the fabric and without involving special equipment or techniques. The possibility of developing such a material has been of great interest to the textile industry ever since the hydrophobic properties of the silicones became apparent. A great deal of research work has been done during the past few years to realize that possibility.

Such a silicone product has been developed and is now in the early stages of commercial testing. This silicone fluid which we designate as DC 1107 has the water repellent properties and general charatceristics of the other fluid silicones. It is different in that it is reactive toward organic fibers and its hydrophobing properties are fully developed by heating for a short time at temperatures which may be applied to such fabrics as wool, rayon, nylon and cotton. It contains no harmful acids or alkalies and is not injurious to rubber or corrosive to metals. Experimental mill runs have shown that conventional equipment can be used.

Since a solvent solution would be costly and hazardous, research work was directed toward the development of a water emulsion. This emulsion can be easily prepared at the mill in concentrated form and diluted with water before being applied.

A two to four per cent emulsion of the DC 1107 Fluid is applied to the fabric by padding and fixed by heating for 5 minutes at 300° F. This gives excellent water repellency to wools, rayons, nylons and cottons. This silicone treatment has very good resistance to dry cleaning and washing. It gives the fabric a soft, full hand and excellent draping properties.

Samples of silicone treated wool serge were tested for dynamic absorption[20] before and after laundering, and dry cleaning. These values and comparable data on untreated wool serge are plotted in Fig. 5. Even after laundering, the silicone treated wool serge samples absorbed less water after 30 minutes than any of the untreated samples absorbed in 10 minutes. The excellent solvent resistance of the

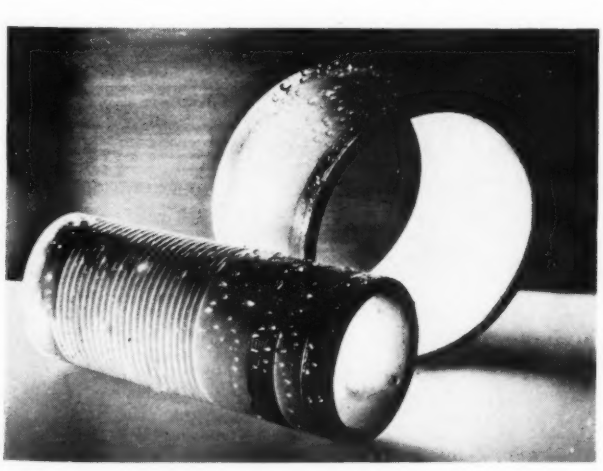

*Figure 3*

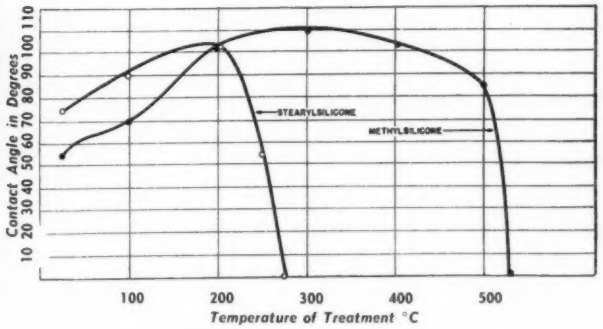


*Figure 4. Contact Angles Soda Lime Glass Treated with Silicones (.02% in Benzene Solution)*

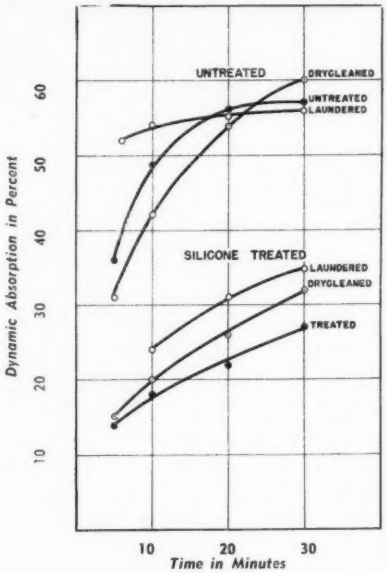


*Figure 5*

*Dynamic Absorption Values on Silicone Treated Wool Serge*

DC 1107 treatment is indicated by the small increase in absorption values after dry cleaning.

After laundering[21], the silicone treated wool serge had about twice the water repellency of untreated samples as measured by these dynamic absorption values. These data indicate that the water repellency of silicone treated wool serge after laundering is comparable to that retained by the better commercial finishes.

The spray ratings of silicone treated nylon, rayons and cottons before and after one, two and three successive launderings are given in Fig. 6. In every case the initial spray rating is 100, indicating that this silicone treatment gives high water repellency to many fabrics. Laundering for 45 minutes in water containing 0.5% soap and 0.2% sodium carbonate at 140° F. has little if any effect on the spray ratings of most of the treated fabrics. After three launderings, the spray ratings were still high, ranging from 70 to 80 except in the case of cotton twill.

The data presented here are limited because the DC 1107 treatment for organic fabrics is a very new development. Much work remains to be done. We feel justified, however, in giving you some of the preliminary test data in view of the keen interest you have shown in the development of a silicone material which might provide a more durable and more effective water repellent finish for textiles. All of the testing done to date indicates that the silicone treatment briefly described in this paper merits careful consideration as a durable water repellent finish.

## Summary

Organosilicon oxide polymers, commonly known as silicones, are built on an inorganic skeleton of alternate silicon and oxygen atoms similar to that found in quartz, glass, mica and the mineral silicates. This exceptionally stable framework accounts for the thermal stability, chemical resistance, and excellent electrical properties of the silicones. The addition of various organic radicals to the silicon atoms imparts properties of water repellency and lubricity, and gives the ease of handling usually associated with the less stable organic polymers.

The silicones are produced in the form of fluids, greases and compounds, resins and varnishes, and rubber-like materials. Certain silicone resins and varnishes have made possible a new class of heat and moisture resistant electrical insulation. Others are used for bonding inorganic fabrics in the production of rigid laminations. Still others are used as vehicles for heat and weather resistant protective coatings.

A silicone compound kills foam in a wide variety of aqueous systems. Silicones with elastic properties are not affected by continuous exposure to temperatures ranging from —70° F. to 300° F. Silicone oils and greases lubricate bearings, conveyor chains and other machinery operating at high temperatures. Other fluids are used as treatments for glass and ceramic surfaces.

A new silicone fluid shows promise as a very effective, durable water repellent treatment for textiles.

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[13] Silicones as Lubricants, T. A. Kauppi and W. W. Pedersen, Tech. Sec., National Petroleum News (Dec. 5, 1945); S.A.E. Journ. (March 1946)

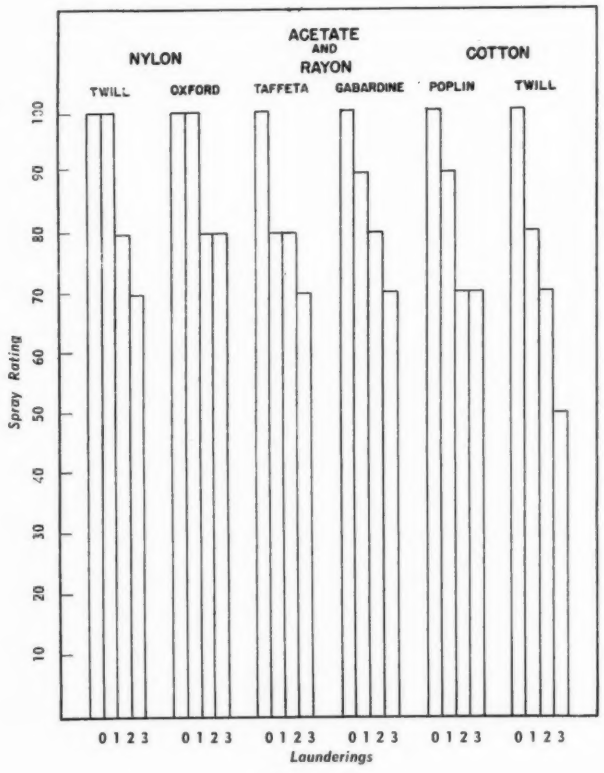


*Figure 6*

*Laundering Resistance of DC 1107 Treated Fabrics.*

[14] DC Valve Seal A and DC Valve Seal B, Dow Corning Corp. (revised Nov. 1947)
[15] DC Antifoam A, Dow Corning Corp. (Feb. 1947)
[16] Silastic Facts No. 5, Dow Corning Corp. (Nov. 1947)
[17] (a) Dow Corning Fluids, Dow Corning Corporation (July 1944) (b) A Comparative Study of G-E Silicone Oils and Petroleum Oils, Resin and Insulating Materials Div., General Electric Co. May 1945)
[18] O. K. Johannson and J. J. Torok, Proc. Inst. Radio Engrs., 30, No. 5 (May 1946)
[19] M. J. Hunter, A. J. Barry, M. S. Gordon, J. F. Hyde and R. S. Heidenreich, Ind. and Eng. Chem. *39*, 1389-1401 (Nov. 1947)
[20] Federal Specification CCC-T-191A and Supplement.
[21] U. S. Army Specification 100-48

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*Chairman Dahlen:* I am sure that this excellent paper has provoked much thought and has been of great interest to you. Probably many of you have questions to ask of our speaker. However, to be sure that the meeting keeps to its time schedule, we shall defer all questions and discussion until after completion of the three formal papers. We then shall use any remaining time for questions and discussion.

Probably no unit process in the textile field is more important and has more aspects than that of drying, the presumably simple process of removing water from textile fibers. Most of you mill men realize that this is a very complex subject and that we need far more fundamental information and practical approaches to this subject than are available today. I am sure that you will all welcome a review of our present fundamental information on this subject and an interpretation of this information in terms of practical application.

We are exceedingly fortunate in having as our speaker, Dr. Albert C. Walker of the Bell Telephone Laboratories. Dr. Walker is a physical chemist, who attended the University of Colorado, received his Bachelor's degree from M.I.T. and his Ph.D. degree from Yale in 1923. He has been associated with the Bell Telephone Laboratories ever since receiving his Doctorate.

A great deal of his research has been devoted to the physical chemistry of textiles and fibers, the moisture behavior of textiles, and the drying of textiles. Remembering the very stringent requirements of fiber-insulated telephone lines and cables, as well as the intricate equipment of the communications industry, we can be sure that Dr. Walker has been confronted with everything from the difficult to the impossible. He has served as a Director of the Textile Research Institute and has been a guiding light on its basic researches on the moisture relationships of textile fibers. Much of the credit for the excellent papers published by that Institute on this subject belongs to Dr. Walker.

It therefore is a very great pleasure for me to introduce Dr. Walker, who will speak to us on the subject, "The Textile Drying Process."

## *General Technical Session—*

# The Textile Drying Process*

A. C. WALKER
*Bell Telephone Laboratories*

IN RECENT years, vast changes have taken place in the textile industry. One important development has been the use of synthetic resins in attaching pigment colors to fabrics, another the application of durable water-repellent finishes. These are but two examples of numerous treatments which require heat to set the resins, vaporize solvents and volatilize by-products of chemical reactions. Thus heat is used for other purposes than the simple removal of moisture. It has been suggested[1] that textile drying be defined as the removal of any volatile substance from a fabric or fiber by means of heat energy. A recent survey[2] emphasizes the need for more accurate operating data on drying equipment and for more research on fundamentals of drying.

At least one step in the manufacture of many industrial products involves drying, and it seems reasonable to term this operation a common denominator process. Because of its basic importance, and increasing complication, drying of textiles deserves continual study from widely different points of view. Therefore, no single discussion of this subject can be truly comprehensive. The purpose of this paper is to review the subject of moisture removal in the light of recent contributions on moisture relations, and to consider the possible advantages of heat application and drying by other methods than those which have been in general use in the industry. Also an example will be given of the development of a practical method of vacuum drying of textiles, which was based on considerations of fiber structure and the location of moisture in the internal surfaces of the fiber. This treatment of the subject is submitted in the hope that it will contribute to a better understanding of the problem of moisture removal, and to be helpful as well in relation to the broader definition of drying—the removal of any volatile substance.

The equilibrium moisture content of a material affects both the rate and extent of drying. Therefore, it is important to know the moisture relations of textiles. Most of the outstanding work on this subject has been published within the past twenty-five years[3], and is reflected in the marked improvements made in textile drying methods in the past two decades.

Just a year ago, an effective generalized correlation was published[4] on the moisture relations of a variety of fibers. The importance of this contribution cannot be stressed too highly. The Othmer chart method, on which this correlation depends, provides a means of obtaining reliable data on the moisture content-relative humidity relations of a textile or other adsorbent material, over a range of temperature, and from only a few careful measurements. This study was initiated in an effort to provide a better means of correcting and extending the Wiegerink[5]

* Presented at General Technical Session, National Convention, Chicago, October 24, 1947.

[1] C. N. Rabold, Amer. Dyestuff Rep., March 12, page 108, (1945).

[2] S. J. Friedman—J. Ind. Eng. Chem.,*39*, (1947), page 39 of Unit Operations Review.

[3] The classic researches of Williams, and Urquhart and Williams, dating from about 1924, probably mark the beginning of this period.

[4] J. C. Whitwell and R. K. Toner, Textile Research, *16*, 255, (1946).

[5] J. G. Wiegerink, RP 1304, Jour. of Research, N.B.S., *24*, June (1940).

data on a number of textile fibers. A previous effort to provide some simplified linear relations[6] based on these data which could be of practical use in industry, involved some admittedly imperfect assumptions, and it is gratifying to have the data now presented in a form less dependent on such limitations. This contribution of Whitwell and Toner[6], together with some additional studies by Wiegerink[7] on the effects of heat and humidity on the properties of textile yarns, now makes it possible to form practical estimates of limiting conditions of temperature, humidity, and time of exposure on the drying of textiles with minimum damage.

As a corrollary, it is entirely reasonable to propose that the application of the Othmer plot method to data on the sorption of other volatile substances than water in textiles may be equally useful in connection with the development of improved finishing processes. Heats of adsorption of suth volatile substances on fibers may be derived from such data and it may be quite advantageous to compare such data with those for water.

The textile industry realizes the need for better drying methods, but the first concern has been, quite naturally, with the economic questions relating to increased rates of drying, and much less with the basic moisture relations, or volatile substance relations. It is a common experience in industry, that many machines are economical and useful although at the time of their design, little was known regarding the principles responsible for their successful operation. For example, the simplest and cheapest form of cloth drying machine, long used in the textile industry, is a set of revolving steam-heated cylinders against which the wet material is pressed. The textile gives up its moisture progressively as it passes along the set, but the method is not suitable for all types of fabrics. Little or no control is provided over cloth dimensions, and cylinder drying imparts a harsh feel and an undesirable stiffness to the fabric as compared with drying by heated air.

Convection drying, that is by means of heated air, introduces further complications. Machines based on this principle utilize a hot air stream to remove moisture from cloth, and although this method is not so efficient as cylinder drying, the commercial advantages with respect to improved finish and dimensional control more than outweigh the extra cost of heat and the increased complication of the machine.

Application of heat by means of radiant energy is basically the oldest means of heat transfer. The earth obtains its heat from the sun in this way, and energy in this form requires no medium for its transfer. Industrially, the radiant energy method of heating employs waves in the near infra-red part of the spectrum. These waves pass readily through gases, glass and some other substances with little energy loss, and they may be reflected almost completely from highly polished surfaces. They originate either at an incandescent refractory surface, from direct gas fires, or from specially designed electric lamps. These waves travel in straight lines from their source and when they strike a material object, such as a textile fabric, this energy is partly reflected, partly transmitted, and partly absorbed. Only the absorbed energy is converted into heat.

There is evidence that infra-red rays penetrate textiles to some extent, and temperatures below the surface are reported to be slightly higher than directly on the surface. However, these differences in temperature are not considered of sufficient significance to change the physical characteristics of the drying operation. Infra-red equipment has been used to supplement existing steam installations for general drying, particularly during the war when maximum output was demanded on existing equipment. The steam load is reduced, and production is speeded up, but only in special circumstances is it likely that this type of drying can be proved economical.

Dielectric drying has been applied successfully in some cases in the textile industry, particularly where dense masses, such as rayon cakes, must be dried as uniformly as possible. When a poor conductor of electricity, such as a textile, is placed between two electrodes and energy of radio frequency is applied, sufficient resistance is offered to the flow of current to convert some of this energy into heat, and this heat is generated at once throughout the entire body of the nonconductor. Thus evaporation of moisture may be promoted through the whole textile mass and drying may be reduced to a matter of minutes rather than hours. Several advantages result, for example, better uniformity of drying and of dye distribution. The method is, however, quite expensive.

Both experimental work and theoretical considerations lead to the conclusions that the conditions for effective moisture removal at any temperature are analogous to those for efficient heat transfer. Also under a given set of conditions, the same amount of energy is required to evaporate one pound of water from any source of energy. Therefore, each drying problem requires individual study, and full consideration of all factors involved. Much confusion has occurred in the textile industry because of failure to give due regard to these matters. It is appropriate here to cite one authority[8]: "One of the most important points in the design of drying apparatus and the control of drying operations is the provision for adequate and uniform air velocity past the material. In the drying of thin sheets, a reasonably high velocity is desirable to secure most rapid evaporation. In the case of thicker sheets, high velocity does not help so much because the major resistance to evaporation in such cases is the diffusion of moisture from the interior of the material to the surface."

There must be an effective scrubbing action between air and cloth to reduce the thickness of the moisture or dead air layer on the surface. Passing air through the cloth gives intimate contact, but only a small amount may be passed in this way. A commercial attempt to apply this principle is that of dividing the air stream into small high velocity jets, closely spaced, which impinge on the cloth at close range.

In dense, moisture-absorbing structures, water can be removed within a reasonable time by the application of vacuum, but there are some limitations in this method which are not widely recognized or understood. For example, consider an experiment on the drying of a package of wet cotton taken from a dye kier. The package, containing about 200%[9] of water by weight, was placed on a tray in a vacuum oven at 250° F. A vacuum of 1 mm. of mercury was applied. After 16 hours it was found that while most of the interstitial water had been sucked out of the package, this was standing in a dish below the tray, and the interior of the oven was "dripping wet." The package still contained upwards of 50% of water, and it was evident that very little water had been removed from the oven. The experiment was repeated, except that a valve on the opposite side of the oven from the vacuum port was cracked slightly so as to bleed air at a slow rate into the oven during drying. With this air leak, the vacuum was only about one-third of an atmosphere. Despite this poor vacuum, the cotton was completely dried over night. The air flow facilitated heat transfer, reduced the thickness of the stagnant water film on the package surface, and

[6] A. C. Walker, Textile Research, *13*, No. 5, March, page 15, (1943).
[7] J. G. Wiegerink, RP 1337, Jour. of Research, N.B.S., *25*, (1940).

[8] "Principles of Chemical Engineering Practice," 1st Ed., Walker, Lewis and McAdams, page 501.

[9] The water content is usually reduced to about 50% to 60% by centrifuging. However, this experiment was made with a maximum amount of water purposely.

provided a directional flow of both air and water molecules in and through the oven to the vacuum port, rather than movement to the port by diffusion alone.

Most of the water in centrifuged packaged cotton is held in or on the individual fibers, and is not easy to remove. The water must be transported to the outside of the package, an average distance many thousands of times greater, relatively speaking, than that between the internal surfaces of the fibers where most of the water is adsorbed, and the external surfaces of the fibers. It is not surprising that from 12 to 20 hours is required to dry a lot of 500 pounds of cotton in package form, stacked closely on trays in a hurricane dryer, even at 250° F., with air circulation of an imperfect sort only around the peripheral packages.

These considerations pose an interesting question regarding the drying of textiles in the form of dense structures. If dielectric drying is too expensive, can some form of vacuum drying be employed which will take advantage of improved means of heat transfer and air velocity as well? In fact, can these principles be applied within the body of the individual fibers? Simply evacuating an oven containing packages certainly does not accomplish this, and the air leak experiment, while better than simple diffusion, leaves much to be desired.

It is appropriate to recall, at this point, the present picture of the structure of fibers. For example, cellulose either in the form of natural or mercerized cotton, or regenerated cellulose, is considered as being built up of very long chain-like molecules. In certain regions these chains lie parallel with one another in such closely packed, regular formation that they appear to possess many of the properties of crystals. Other regions show no such regularity, and are termed non-crystalline, but some of the single long-chain molecules may run continuously through both alternate crystalline and non-crystalline regions. Thus a fiber may be described as an irreguar flexible net built up of crystalline and non-crystalline regions, with numerous holes where the crystallinity is a minimum, with spaces or crevices in intermediate regions, and with relatively few openings in the highly crystalline regions.[10] X-ray evidence indicates that the highly crystalline regions are relatively inert to chemical attack; they are not penetrated by water, dyestuffs or other reacting substances. However, these may enter into the non-crystalline regions to a greater or less degree, depending upon the density of the structure, and upon the accessibility of holes, cracks or crevices.

[10] Structure of the Rayon Fiber, H. Mark, Nature, *144*, 313 (1939)

Dry cotton will regain not more than about 25% of moisture, by weight, when in equilibrium with a saturated atmosphere. More than twice this amount may be held in the fiber structure when the cotton hair is wet and swollen with liquid water. Consideration of the heat of wetting of dry cellulose and its dielectric constant at various moisture contents, indicate that the first moisture adsorbed by dry cotton is more firmly bound than subsequent amounts. A recent calorimetric study of moisture adsorption in cellulose summarizes these considerations as follows:[11] "Modern concepts of the mechanism of moisture adsorption by cellulose fibers involve three steps: (a) the selective adsorption of water molecules by hydrogen bonding to the hydroxyl groups in the non-crystalline or micellar regions; (b) the adsorption of water into the lattice of the crystalline regions in those cellulose structures exhibiting changes in cellular dimensions with moisture adsorption, e.g. regenerated or mercerized fibers; and (c) capillary condensation or "multiple-layer formation" in those spaces in the cellulose fiber which permit occupancy by additional water molecules."

While some water is held in gross amounts in large holes there is a considerable amount of water existing as multimolecular layers in the internal surfaces. This water is not to be considered as continuous layers of solid water completely filling such spaces or cracks as molecular layers between contiguous cellulose surfaces, but as columns or chains of water molecules, each chain being anchored by hydrogen bonding of the first water molecule to a hydroxyl group in the surface of the cellulose complex. Thus there may be large numbers of small air spaces between these chains, quite analogous to the air spaces between vertical fibers in a pile fabric, like velvet.

Suppose that a package of centrifuged cotton is placed in a vacuum oven operated at 250° F., and the system is suddenly evacuated. Air molecules in the internal surface areas, surrounding the liquid water chains will rush out rapidly because their movement is more or less directed and stimulated by the pressure differences between the applied vacuum and the atmospheric pressure inside the fiber. Water molecules will evaporate from the free ends of the water chains, and these will aid in driving before them much of the air. Water removal will continue at a constantly decreasing rate until the temperature of the wet fiber is reduced to a point where the vapor pressure of the liquid water is substantially equivalent to that of the vacuum being maintained. Further removal of water from this steady vacuum can occur only at very small differential pressures, the diffusion is of a random order, not directed as at first, and eventually a film of water vapor will be built up on the fiber retarding evaporation. Diffusion under such conditions is an extremely slow process. Within the fiber itself the mean free path of the water vapor molecules is greater than the distances across the pores or cracks and there is little or no directional effect to help diffusion.

[11] Calorimetric Investigations of Moisture in Textile Fibers, F. C. Magne, H. J. Portas, and Helmut Wakeham, J.A.C.S., *69*, 1896, (1947).

Water may evaporate from chains situated near the entrances of pores or other openings in the structure more readily than from chains more deeply buried in such air spaces. Therefore there may be an uneven distribution of chain length between pore entrances and interiors.

If dry air is now admitted to the oven it is heated by contact with the oven walls and the fiber temperature is raised by conduction and convection. This air penetrates rapidly to all of the free internal spaces in the fiber and mingles freely with the relatively small number of vaporized water molecules already there. The air flow into the fiber spaces stirs and agitates the water chains, conceivably assisting in re-distributing liquid water molecules between chains of different lengths, and promoting evaporation as well.

On re-applying the vacuum the first cycle is substantially repeated, differing only in the amount of water which may evaporate from the chains. This action, which may be described as a sort of flushing, is aided to the degree to which the fiber may be re-heated during the air admission cycle.

Under these conditions an air flow takes place over the internal surfaces, similar in character to that which occurs at textile surfaces in ordinary drying practice using air streams or jets. The magnitude of the air velocity within the fiber is, of course much less than on the outer surface and the duration of the effect is much shorter. The fact that it takes place at all is important.

This process should prevent excessive drying in the more accessible areas at the expense of water more deeply buried in the structure. Uneven drying may result in complete removal of water from hydroxyl groups at the entrance to fine pores and these hydroxyl groups may then be attracted to others on contiguous surfaces thus closing the pores and trapping considerable moisture within them. Such a conception helps to explain permanent alterations in the fiber structure when it is

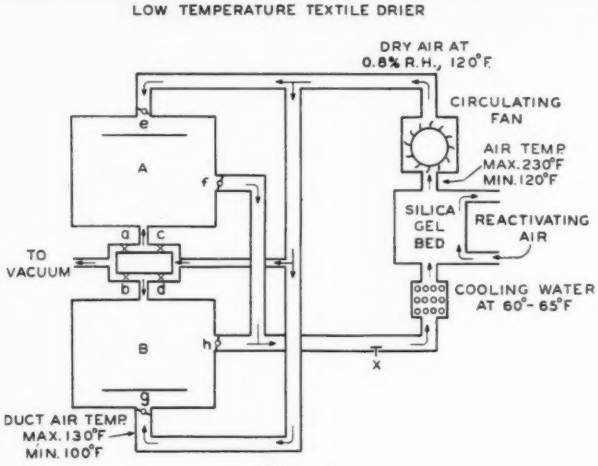


*Figure 1*

dried to a moisture content below that corresponding to the monomolecular layer.

Alternate applications of vacuum and breaking of the vacuum with dry air, coupled with vigorous air circulation through the oven during the air cycle at atmospheric presure is the basis of a textile dryer developed by the Western Electric Co., for drying textile insulated copper conductors wound on large drums. A schematic diagram of the esesntial features of this dryer is shown in Figure 1. To prevent oxidation of the copper it was essential that the textile be dried at a relatively low temperature, not exceeding 120° F. This condition made it difficult to remove moisture by conventional equipment.

In Figure 1, ovens A and B are connected by a vacuum manifold to a high capacity vacuum pump with valves a, b, c and d suitably placed in the manifold. Each oven is connected to a metal duct system through which may be circulated, as desired, a large volume of dry air at substantially atmospheric pressure. The duct system is connected to a silica gel absorber capable of maintaining this air at somewhat less than 1% relative humidity at 120° F. Cooling coils are provided to control the air temperature and a large circulating fan is employed to drive the air at high velocity through the ducts, oven and gel bed. Moisture taken from the textile is absorbed in the gel continuously and the gel is reactivated periodically by means of a steamheated reactivating system indicated in the diagram.

The apparatus operates as follows:

After filling both ovens with the material to be dried, the doors are sealed and valve a is opened so that oven A may be evacuated to about 2" of Hg pressure. Valves e, f, g and h are flap type closures which cannot be opened until the oven is at atmospheric pressure. Therefore, during the removal of air from A valves c, e and f are closed, and valves d, g, and h are opened by a synchronized mechanism so that dry air is circulated through B. After 15 minutes valve a is closed and c is opened permitting dry air to flow into A from the manifold connection to the duct. To prevent the duct and silica gel system from falling below atmospheric pressure a bleed valve x opens until A reaches atmospheric pressure. Then valves e and f open and dry air is circulated at high velocity through A. At the same time valve d is closed and b opened.

Valves g and h close synchronously and the oven B is evacuated. Repetition of this alternating cycle at 15 minute intervals results in rapid and uniform drying of this textile to an extremely low moisture content.

* * * * *

*Chairman Dahlen:* The third paper on this morning's program is devoted to another phase of our general interests. The past few years have witnessed tremendous growth of the plastic and resin sciences and industries. From this work have come new synthetic fibers which are really oriented plastics. They are finding their place in many textile products. We also have seen the growth of unwoven fabrics, produced by bonding together textile fibers by the use of many types of resins and the application of new techniques. Still further, we have seen plastic sheeting materials and lamination of plastic sheetings with textile fabrics and other materials to produce many new and valuable products. All of these developments are having an important impact on the present and future textile industry. In part, they represent new competition for textiles; but they also offer many new opportunities for the development and exploitation of new and profitable textile products.

Our committee was confident that you would all welcome an up-to-date discussion of these new materials. We have been most fortunate in securing C. W. Patton, Manager of the Coating and Adhesive Materials Division of the Bakelite Corporation, a unit of Union Carbide and Carbon Corporation, to present this subject.

Mr. Patton is a graduate of Marietta College. In 1932, immediately upon receiving his degree, he began research work in the laboratories of Carbide and Carbon Chemicals Corporation, another unit of Union Carbide. After several years, he went to the Mellon Institute under the fellowship of the Carbide and Carbon organization, and after three years of this activity he entered into development and sales work on vinyl resins. When Union Carbide and Carbon acquired the Bakelite Corporation and merged all plastic activities, he was appointed Manager of their Coating and Adhesive Materials Division. This division includes in its activities, resins for yarn and cast films.

Our speaker is well known to the New York Section. He has presented papers before this Section and was Chairman of their Intersectional Contest Committee a few years back. From this very brief introduction, I am sure that you will agree with me that Mr. Patton is extremely well informed on plastics and related products and their relation to the textile industry. It is a pleasure to introduce Mr. Patton, who will speak on "Plastic Materials that Supplement or Supplant Textiles."

*(See next page)*

## CALENDAR

PHILADELPHIA SECTION

Meetings: January 9, 1948, February 27, April 9, May 14 (Kugler's, Philadelphia).

NEW YORK SECTION

Meeting: January 23, 1948 and March 12 (Hotel New Yorker, N. Y.).

PIEDMONT SECTION

Meetings: February 7, 1948 (Poinsett Hotel, Greenville, S. C.); April 3, 1948 (Robt. E. Lee Hotel, Winston-Salem, N. C.); June 18, 1948 (Ocean Forest Hotel, Myrtle Beach, S. C.).

RESEARCH SUB-COMMITTEES

Meetings: January 8, 1948 (Downtown Athletic Club, New York).

COUNCIL

Meeting: January 9, 1948 (Downtown Athletic Club, New York).

RESEARCH COMMITTEE

Meeting: January 9, 1948 (Downtown Athletic Club).

## General Technical Session—

# Plastic Materials

## That Supplement or Supplant Textiles*

C. W. PATTON
*Bakelite Corporation*

YOUR National Technical Program Committee Chairman, Dr. Miles Dahlen, by the very act of suggesting the general nature of this paper has typified the attitude in the textile industry in general, and in the AATCC in particular, of constantly surveying the possibilities of plastic materials in the textile field.

It is certainly an alert and progressive attitude, and a logical one. As a matter of fact, the record of the textile industry shows there are manifold instances where textile equipment, processes, distribution systems, and general know-how can be profitably used in connection with plastic materials. Obviously, the writer would like to aid and abet such tendencies.

Numerous articles have appeared in our official publication—the AMERICAN DYESTUFF REPORTER — concerning specific technical aspects of plastics in the textile field. The writer knows of at least three on uses, chemical composition, methods of manufacture, testing guides, etc., having had to work on said three in most cases to beat a deadline. ("Application of Vinyl Resins and Plastics to Textiles"—AMERICAN DYESTUFF REPORTER March 15, 1943. "A Study of Textile Fibers and Fabric Construction Influencing Adhesion of Textile-Plastic Combinations"—AMERICAN DYESTUFF REPORTER November 22, 1943. "Unsupported Films and Sheeting"—AMERICAN DYESTUFF REPORTER June 3, 1946.) Similarly, such articles are available in various Plastics publications. Although the members of AATCC, as the name indicates, are concerned with highly technical matters, there are and will continue to be numerous problems and decisions to be considered where it is necessary to have a broader view of the field than we normally require in our day to day activities. In view of this, the writer believes this is an opportune time to get an over-all picture of the plastics-textile field in as technical a manner as possible.

The accompanying condensed outline has been prepared to present a sort of thumb-nail sketch of plastics in the textile industry. By dividing the items into two main groups, namely, those in which plastics supplement textile items, and plastics items which supplant textiles, it is hoped to introduce at least some semblance of a classification to aid our thinking on the matter. Moreover, it gives a quick picture of supplementary and competitive aspects that are of vital interest to both the textile and plastic fields. It may even provide some clue as to future trends.

There are admittedly some glowing generalities and conceivably some glaring ommissions in the outline but insofar as possible these will be covered in more detail as each item is discussed.

Items (a), (Misc. Textile Finishes) and (b), (Textile inks), of Group I are undoubtedly so familiar to members of the AATCC as to require no further comment.

Likewise in Group I for item (c) (Synthetic fibers). However, it may be of interest to some that the extraordinary chemical resistance and inertness of vinyl chloride-acetate resin fibers makes possible fabrics for filtering acids, alkalies, food and pharmaceutical products—which, to that extent, gains new markets for textile products.

Item (d), (Misc. base materials for laminating and molding, may be somewhat misplaced in Group I as it is literally an example of how textiles supplement plastics by providing highly desirable reinforcing strength. But since the weight ratio of textile material to plastic is not inconsiderable, it has been included in the group.

In item (e) (woven monofil fabrics), the plot thickens, so to speak, and we have a product the base material for which, namely a plastic monofilament (or monofil, for short) is an outgrowth of the plastic extrusion industry. Developmentally speaking, extruders of tubing and particularly rods, kept making smaller and smaller diameter materials and when they got down to the neighborhood of ten one-thousandths of an inch in diameter, the textile industry rapidly took over and began weaving fabrics of them. Wire-cloth looms were also used but the high filling tension caused difficulties. Hence I believe it is safe to say that the bulk of such weaving is done on modified textile looms.

The chemical and abrasion-resistance of these fabrics in addition to their peculiar "slip" or surface-smoothness characteristics and ease of cleaning, have rapidly projected them into the indicated fields.

Item (f), (impregnated cotton, asbestos, and fiberglass tapes and fabrics) is a highly specialized field which most of us do not ordinarily come in contact with unless confronted with the problem of ripping apart some electric power transmission cable or removing the innards of an electric motor for repairs. The use of plastics has contributed such properties as electrical-fire, and moisture-resistance and in particular the silicone resins with fiberglass cloth have imparted a high degree of temperature resistance. By virtue of this, motors can operate at elevated temperatures which in turn permits greater efficiency and smaller size of the power unit, and higher speeds of rotation.

Unwoven fabrics, Item (g), particularly where the end-use for the material involves no further treatment such as impregnation or coating, may be strengthened by having the fibers bonded laterally with a vinyl acetate resin adhesive. For products for a single or a relatively small number of uses before being discarded, the economies of non-weaving are obvious. These fabrics, when impregnated, give a product of unique "hand" or resilience properties not readily attainable with conventional woven fabrics. These properties, plus the abrasion and soil-resistant properties of the plastic coating, which is often applied over the impregnated material, make them of value in shoe manufacture.

The application of the lines of adhesives to unwoven fabrics may be regarded as primarily a textile operation. I believe in most, if not all cases, a water-base material is used, hence it fits in with normal textile operations. The coatings are applied, in general, by spreader or rubber-type calenders, thus the process may be considered an off-shoot of the so-called "proofing" industry.

By coating yarns, Item (h) (coated

* Presented at General Technical Session, National Convention, Chicago, October 24, 1947.

## CONDENSED OUTLINE OF PLASTICS IN THE TEXTILE FIELD

| | PRODUCT | TYPE OF PLASTIC (or Resin) INVOLVED | GENERAL FIELD of PROCESSING | END-USES |
|---|---|---|---|---|
| GROUP I (Items wherein plastics supplement textiles) | (a) Misc. Textile Finishes. | urea-formaldehyde<br>melamine<br>vinyl acetate<br>vinyl alcohol<br>styrene copolymers | Conventional textile. Padding, etc. | Shrink, crush, and wrinkle-proofing. Weave stabilization, etc. |
| | (b) Textile inks. | acrylate<br>cellulosic<br>vinyls | Conventional textile. Roller, screen-printing. | General textile decoration. "Pad dyeing." |
| | (c) Synthetic fibers. | cellulose acetate<br>polyamides<br>vinyl chloride-acetate<br>vinyl chloride-acrylo-nitrile, vinylidene chloride | Conventional textile. | General fabric uses — specialty filter fabrics, collar interliners, etc. |
| | (d) Misc. base materials for laminating and molding. | phenolics<br>polyesters<br>urea-formaldehyde<br>melamines | Plastic (except for textiles used for re-inforcement) | Architectural, mechanical, electrical. Table tops to timing gears. |
| | (e) Woven monofil fabrics. | vinylidene chloride<br>ethylene (experimentally) | Plastic — for production of monofils.<br>Textile—for weaving. | Automotive, transportation, theatre, etc. seat-covers. Handbags. Insect screening. |
| | (f) Impregnated cotton, asbestos, and fiberglass tapes and fabrics. | vinyl chloride-acetate<br>cellulose<br>silicones | Textile—(weaving)<br>Plastic—(impregnation) | Electrical insulation. Wiring, motors. |
| | (g) Unwoven fabrics. (plain, rubber-impregnated, coated) | vinyl acetate (binder)<br>vinyl chloride-acetate<br>vinyl chloride<br>cellulose | Textile (for base "fabric")<br>Plastic<br>Rubber | Disposable cloths — diaper linings, bibs, shoe-wiping cloths, etc. Shoe-lining materials, belts, handbags, book-covers, etc. |
| | (h) Coated yarns (including twisted paper yarns) | cellulose (nitrate, aceto-butyrate)<br>vinyl chloride-acetate | Mixture of plastic and textile processes.<br>Possibly electrical for those film-wrapped. | Similar to uses for monofil fabrics. |
| | (i) Coated fabrics<br><br><br>glazed chintz) | vinyl chloride-acetate<br>vinyl chloride<br>vinyl butyral<br>cellulose nitrate<br>(water-base materials) | Plastic<br>Rubber<br>(except for base fabric)<br><br>(textile) | Rainwear mattress covers, upholstery, shoes, hospital sheeting, wall coverings, table cloths. (Upholstery, etc.) |
| GROUP II (Items wherein plastics supplant textiles) | (a) Tea-bag paper | (viscose rayon)<br>vinyl chloride-acetate | Textile—for staple fibers (scraps for other type papers)<br>Paper—for base material | As indicated by product name. |
| | (b) Coated latex-impregnated papers | vinyl chloride-acetate<br>vinyl chloride<br>cellulosic | Paper and Plastic | Table cloths, wall coverings, children's bibs, book, luggage, radio coverings. |
| | (c) Plastic sheeting (10 mils thick and up) | vinyl chloride-acetate<br>vinyl chloride | Plastic<br><br>(textile-printing) | Upholstery, ladies' and children's shoe-uppers, handbags. |
| | (d) Plastic films (approx. 4 mils thick) | vinyl chloride<br>acetate<br>vinyl chloride<br>ethylene (limited) | Plastic<br><br>(Textile—printing and flocking) | Rainwear, shower curtains, window curtains and drapes, table cloths, bedspreads, garment bags. |

yarns), it is possible to retain to a considerable degree the handling and hand properties of the base yarns involved. Likewise, proper selection of the base yarn can exert control over the elongation and resistance to elevated temperatures as compared to straight monofils. While the uses are, in general, closely related to those for monofils, the above differences in properties can be significant for many uses. It is difficult to draw a fine line of distinction as to whether the process is a variation of the copper magnet wire enamelling process used by the electrical industry, or warp slashing of the textile industry. The method of manufacture which involves wrapping the yarn with a heat-sealable film appears to have been derived from the electrical insulation industry. As indicated in the accompanying outline, uses for coated yarns are similar to monofil uses.

While the products of the coated fabrics industry, Item (i), are quite well known by members of AATCC, it is worth emphasizing that the durable properties of the vinyl resins have permitted production of greatly improved materials, which in turn is increasing the outlet for the base fabrics involved. New techniques for the coater which involve the use of vinyl resins dispersed, not dissolved, in low-cost volatile liquids or in some instances solely non-volatile plasticizing liquids permit economies heretofore unattainable. This also in turn broadens the base for textile markets. In general, the plastic or resinous-base coatings are applied by knife spreader or rubber-type calenders which are not normally found in textile factories. Glazed chintz is in effect a contradiction to the general statement on processing of coated fabrics because it has been produced for years in conventional textile finishing plants. This is probably due to the fact that until relatively recently only water-base coatings were used.

Having hit the high spots on the first phase of the subject, we shall more closely scrutinize the products in Group II because these directly replace textiles for certain end-uses.

Item (a) (Tea-bag paper), is produced from viscose rayon and vinyl chloride-acetate resin staple fibers in paper-making equipment. There is no need for sizing of any type in this process hence a source of undesirable taste is avoided. Another big advantage over a fabric bag is that the latter has to be sewn or stapled together whereas the paper containing vinyl chloride-acetate resin staple fiber can be fabricated into bags and filled at very high speeds by simple heat-sealing operations. This item does not rank high as a competitive threat to the vast textile industry, particularly since the bulk of the material involved is viscose rayon staple fiber, a textile product. However, the use of a thermoplastic fiber illustrates a principle, and is being investigated in felt manufacture. It may also be of interest in the manufacture of other types of non-woven fabrics. We might also add, in a lighter vein, that this instance of the paper industry competing with the textile industry is long over-due since the textile industry has for many many years been exacting a tithe from the paper industry for fabric scraps and waste needed to make rag papers!

Another product which directly replaces fabrics for some uses is Item (b), plastic coated latex-impregnated paper. The latex impregnation gives improved tear-resistance and a modicum of desirable draping or hand characteristics. The plastic coating provides additional strength and above all a wear-resistant, impervious, readily-cleanable surface. This product has found a wide and ever-increasing consumer acceptance for table and wall coverings. Also some progress is being made with its fabrication into such items as children's bibs. The material when colored and embossed serves as a durable outer covering for low-cost luggage, for note-books, miscellaneous novelty packages or containers, etc. All operations involved in manufacture of the material, except possibly fabrication of certain finished items, lie substantially within the paper and plastic fields.

Plastic sheeting, Item (c) in Group II, is a substantially all-plastic product, both as regards its composition and method of manufacture. Incidentally, the term sheeting indicates a flexible material of 10 mils (10 thousandths of an inch) or more, in thickness. It may be produced on heavy duty rubber type calenders or by a dispersion resin casting process, although, in general, the calendering process greatly prevails for thicknesses of 10 mils and up. It is produced in a wide range of flexibilities, colors, and surface textures. More recent methods of color treatment by printing the "reverse" side of colorless sheeting harks back to the textile industry's treatment of greige goods. It should be of interest to the textile industry that although the plastic sheetings compete directly with textiles, there are many instances where it is desidable to back the sheeting with fabric—for example, where is is necessary to have strong, durable sewn seams and where fabricating would result in placing the sheeting under continuous relatively high tension. The permanent tension does not ncessarily harm the sheeting but may be undesirable, as in the case of shoe toes where underlying structures would be deformed.

The last item (d), plastic film, is unquestionably so well known to members of the AATCC as to warrant practically no further comment. But in order to round out the picture of plastics in the textile field, attention should be called to the properties such as mildew—water—flame—soil—and wear-resistance, that make it a desirable product for the indicated end-uses. Potential decorative, or styling, and economic possibilities should not be overlooked. For example, the material can be covered with flock to produce a type of velvet, and improved printing processes, possibly evolved from textile printing, indicate advances on both scores. It is interesting to note that the heat-sealability of plastic film has introduced a new technique of fabrication to the textile field. Possibly adaptation of this process to fabrication of conventional fabrics awaits only development of a suitable adhesive material. Preliminary work on plasticizer dispersions of vinyl chloride-acetate resin has shown some promise. This type of development would further complicate the textile-plastic field. For example, a plastic supplanting textiles would be responsible for a material that supplements textiles.

As mentioned at the beginning, specific technical details have been presented in the past so possibly the question period which follows will serve to uncover those missed in the past.

To sketch the over-all pictures—

We see (1) plastics or resins are obviously a vital part of the textile field.

(2) Products involving plastics replace textiles in certain fields.

(2a) Even in this latter instance, several textile processes are involved.

(3) The properties which plastics contribute in both cases, and this at the expense of over-simplification and probably some notable omissions, are:

(a) Inertness—resistance to electrical conductivity, mildew soiling, burning, water, general chemical attack, and abrasion.

(b) Impermeability, in the case of coatings, sheetings, and films.

(c) Certain economies (pad dyeing, durability, speed of production).

(4) Textiles have certain very outstanding properties such as—

(a) porosity
(b) absorbency
(c) draping, or hand, qualities
(d) tear-resistance
(e) consumer acceptance of texture

(5) Plastics may predominate in certain so-called textile end-uses which may have the effect of freeing strictly textile activities along such lines and permitting the

textile industry to concentrate on, and take full advantage of, basic fabric properties. (For example, plastic film basically fulfills the requirements of a low-cost, area-covering, waterproof barrier between shower and bathroom. A fabric's properties of porosity, absorbency, and to some extent hand qualities, are virtually wasted or even a detriment, hence the textile skill, equipment and raw materials may be more advantageously concentrated on the towels used in said bathroom, or clothing where the basic properties of textiles are absolutely imperative.)

(6) Textiles and plastics are literally inextricably intertwined. It is hoped this presentation will aid those in both the textile and plastic fields to an increasingly profitable utilization of the inter-relationships.

—♦ ♦—

## Meeting Dates, Piedmont Section

THE following dates and places have been reserved for meetings of the Piedmont Section for 1948. The Fall meeting will be replaced by the National Convention.

February 7th—Poinsett Hotel, Greenville, S. C.

April 3rd—Robert E. Lee Hotel, Winston-Salem, N. C.

June 18th—Ocean Forest Hotel, Myrtle Beach, S. C.

—♦ ♦—

## Meeting, Lowell Textile Institute Student Chapter

A MEETING was called to order on November 4, 1947 by Chairman Bianco and the minutes of the last meeting were read and accepted.

The Treasurer's report was then read and accepted.

Mr. Kenniston, Chairman of the Program Committee, submitted a report for his committee. Programs already completed include:

October 21—The Shell Oil Company film "Prospecting for Petroleum" was presented to about seventy students in Southwick Hall at 4:00 P.M.

October 28—The first in a series of seminar programs was held at 7:30 P.M. Doctor Harold W. Stiegler, Director of Research for AATCC, spoke on "Your Ideas Won't Work Unless You Do—The Challenge of Research to the Textile Chemist". The program, which also included a question period and refreshments, was enjoyed by thirty-five students and guests.

November 13—The American Viscose Corporation film "How Rayon is Made" was shown in Southwick Hall at 4:15 P.M.

November 25—Frank V. O'Neil, Director of the Physical Research Laboratory at the Pacific Mills in Lawrence, Mass., addressed the Seminar group at 7:30 P.M. His topic was "Proper Conditions for Color Matching".

December 9—Martin H. Gurley, Jr., Chief of the Textile and Paper Section, Esselen Research Corporation, addressed a Seminar group at 7:30 P.M. His topic was "Manufactured Fibers".

Future programs include:

January 27—Dr. Donald H. Powers, Monsanto Chemical Company, will address a Seminar group at 7:30 P.M. His topic will be "The Role of Synthetic Resins in the Modern Textile Field".

Mr. Kenniston announced that additional programs are being arranged.

A letter from Dr. Robert D. Eddy, Chairman of the Committee on Student Affiliates of the American Chemical Society, was read to the Student Chapter. The letter announced a proposed meeting of Chemistry Students in the New England area to be held in Boston in March or April, and included a questionnaire designed to determine what kind of support such a project would receive. This questionnaire was completed and is being returned to Doctor Eddy.

The motion was then made and accepted that the meeting adjourn.

Respectfully Submitted,
LAWRENCE C. GARDNER
Secretary

—♦ ♦—

## Surface Active Chemicals for Textile Processing*

DR. E. A. ROBINSON

*Technical Director, Industrial Division, Nopco Chemical Company*

CONSIDERABLE work has been done in evaluating the surface characteristics of all types of surface active chemicals as well as raw oil constituents. From an evaluation of approximately 3,000 different chemicals, it has been possible to classify these materials as to the coefficient of friction values they impart to various textile fibers. It is thought that those products which exhibit the lowest coefficient of friction value are the chemicals which penetrate and soften the textile fibers to the least extent. By blending two or more of these chemicals together and applying them to textile fibers, the results indicate that as far as coefficient of friction values are concerned, the materials exhibit additive characteristics. Thus, the oil chemist is able to formulate a product for the processing of textile fibers embodying low and high coefficient of friction constituents which will produce a medium coefficient of friction balanced product possessing many desirable processing features.

In the preparation of soluble oils, many types of surface active chemicals can be employed. Those which exhibit the most popular use are the ones which are soluble in a raw oil and produce a homogeneous mixture. This soluble oil is capable of being added to water to produce stable emulsions or dispersions for application to textile fibers.

All of the soluble oils which have been employed in the textile industry have required the use of a synthetic detergent at some subsequent scouring operation in order to remove these surface constituents. It has now been found that special emulsifying and detergent chemicals can be tailor-made and employed in large quantities in soluble oils. By such a procedure, the soluble oil serves two functions: the first is the proper lubrication of the yarns and fibers and secondly, detergents are not required for the removal of these special soluble oils during the scouring operation.

By applying this new principle, many types of new oils can be prepared which would permit many modifications in the procedure being followed for the processing of textile yarns and fibers.

* Summary of a paper presented before the Rhode Island Section, October 31.

## EMPLOYMENT REGISTER

*This column is open for four insertions per year, per member, without charge. Blanks can be obtained from, and filed with, the Secretary of the Association, Lowell Textile Institute, Lowell, Mass. It is understood that these will be open to inspection by prospective employers, who can obtain further information from the Secretary.*

47-39

*Education:* B.T.C., Lowell Textile Institute, 1936.

*Experience:* Textile chemist, operator of dyeing and finishing equipment, laboratory manager and inspector, manager of dyeing plant.

Age, 35; married, references.

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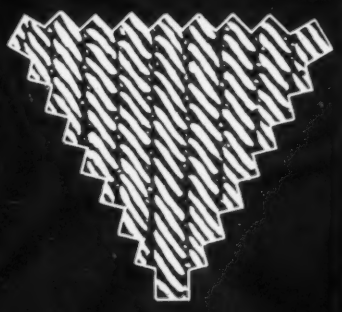

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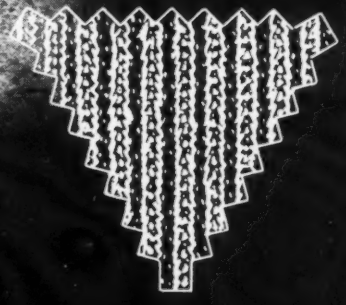

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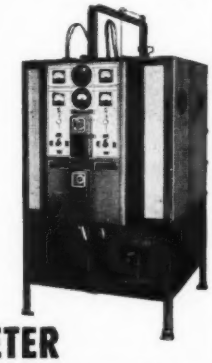

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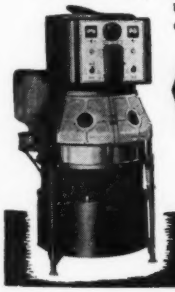

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L&N instruments can be equipped to provide any desired type of automatic pH control, from simple on-off to fully proportioning. The latter is rapidly coming into wide use because it meets every change in acidity or alkalinity of the liquid with a strictly proportional change in the amount of chemical supplied. Because of the extreme accuracy of this balance, pH is held within entirely new limits, and full advantage is taken of the relationship between quality of water or other product, and control of acidity.

If you have a pH recording problem and will outline it, an L&N engineer will be glad to help select equipment to meet your need.

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American Dyestuff Reporter

POSITION WANTED: Chemist-Colorist 33, B.S. 18 years experience napthol, vat, indigosol dyeing and printing. Bleaching, dyeing and finishing. With plastic rayon cellulose research-development background. Interested only in big job that needs good trouble shooter. Can build continuous dye machines. Write Box 360.

WANTED: By Plant doing Bleaching, Mercerizing, Dyeing & Finishing of cotton piece goods, man 25 to 35 years old, with technical training and some experience in plant operations to train for position of assistant to plant superintendent. Write Box 361.

WANTED: Man with sales experience in dyestuffs, soaps and textile specialty field to represent in Canada an old and well established house. Reply to Box 362 stating experience and qualifications. All replies will be kept strictly confidential.

WANTED: Leading Southern Manufacturer invites applications of graduate Chemists having experience in Textile Auxiliaries. Write stating qualifications and experience. Write Box 356.

Technical sales representative for weaving, throwing, knitting and finishing trades; good sales producer and business builder; free to represent one or more small, ethical textile chemical manufacturers; capable of assuming complete sales management duties. Write Box 357.

Chemical firm with good contacts among textile mills and processors can sell resin emulsions of merit, especially creaseproof finishes. Write Box 358.

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TECHNICAL SALES CHEMIST—To develop uses for colloids in food and pharmaceutical fields. Headquarters New York area. B.S. in chemistry with several years technical sales experience. Age 28-38. State experience and salary requirements. Write Box 339.

DYESTUFF CHEMIST—Experienced in textile printing for technical sales and service. Headquarters New York area. Age 28-38. State experience and salary requirements. Write Box 340.

TECHNICAL SALES REPRESENTATIVE: Wanted by well-established sizing specialists to help develop line of textile auxiliaries and finishes. Require: young man, at least 5 to 10 years' experience in wet processing and finishing, good sales personality. Good salary for right man, excellent future. Southeast. Write Box 344.

POSITION WANTED: Superintendent of dyeing, technical college graduate, age 47, 25 years' experience cotton or rayon, skein or package, all types of colors. Excellent references. Would consider sales and demonstration. Write Box 343.

CHEMIST OR CHEMICAL ENGINEER: With firm background in field of textile chemistry, and preferably having some knowledge of resin polymers, for research in evaluation and development of resinous finishes for textiles. Responsible position, salary commensurate with experience. Metropolitan New Jersey. Give full details in first letter. Write Box 345.

Sales Representatives for long established manufacturer of complete line of soap and allied products, for textile, laundry, janitor supply, etc. fields. Will consider full-time salary and bonus or part-time commission. New England and Southern territories open. Write Box 346.

POSITION WANTED: As lady hosiery dyer possessing laboratory research and sales control and dyehouse production. Seventeen years as head dyer for a large hosiery manufacturer in the South. Excellent references as to ability and character. Write Box 348.

POSITION WANTED: Cotton and rayon piece goods finishing control chemist with 23 years' experience. College graduate, able to assume charge of laboratory. Prefer location north of Virginia along seaboard. Write Box 349.

WANTED: A foreman for California plant, experienced in dyeing (fast colors) and finishing cotton, silk, rayon, wool piece goods and yarns. Write Box 353.

RESEARCH CHEMIST: Large textile company offers attractive position in its Research Department to a well trained chemical engineer or chemist, preferably with some experience in the field of textile research. Location Newark, N. J. In reply give age, scholastic background, experience and salary expected. Write Box 359.

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ANTHOMINE: Auxiliary used in all wool dyeing processes to produce a uniform shade and impart a soft pleasing hand to stock, yarn and cloth. Improves exhaustion of dye bath and increases color yield. Very effective for levelling shade of tippy and mixed wools.

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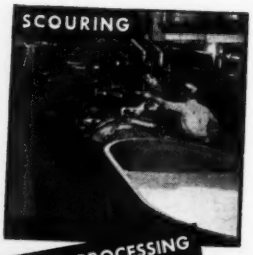


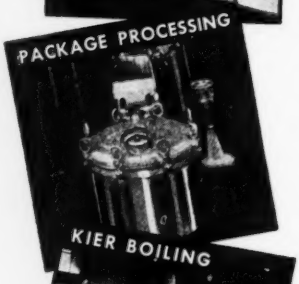

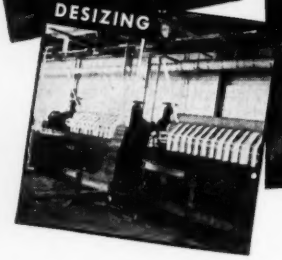

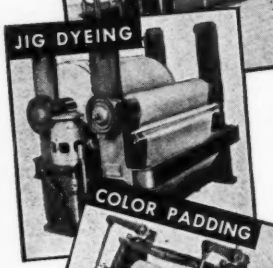


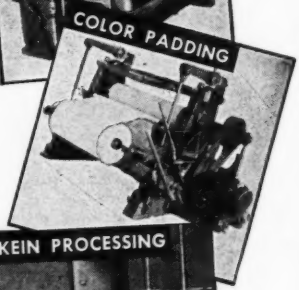


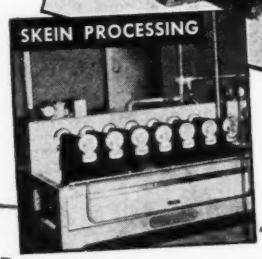


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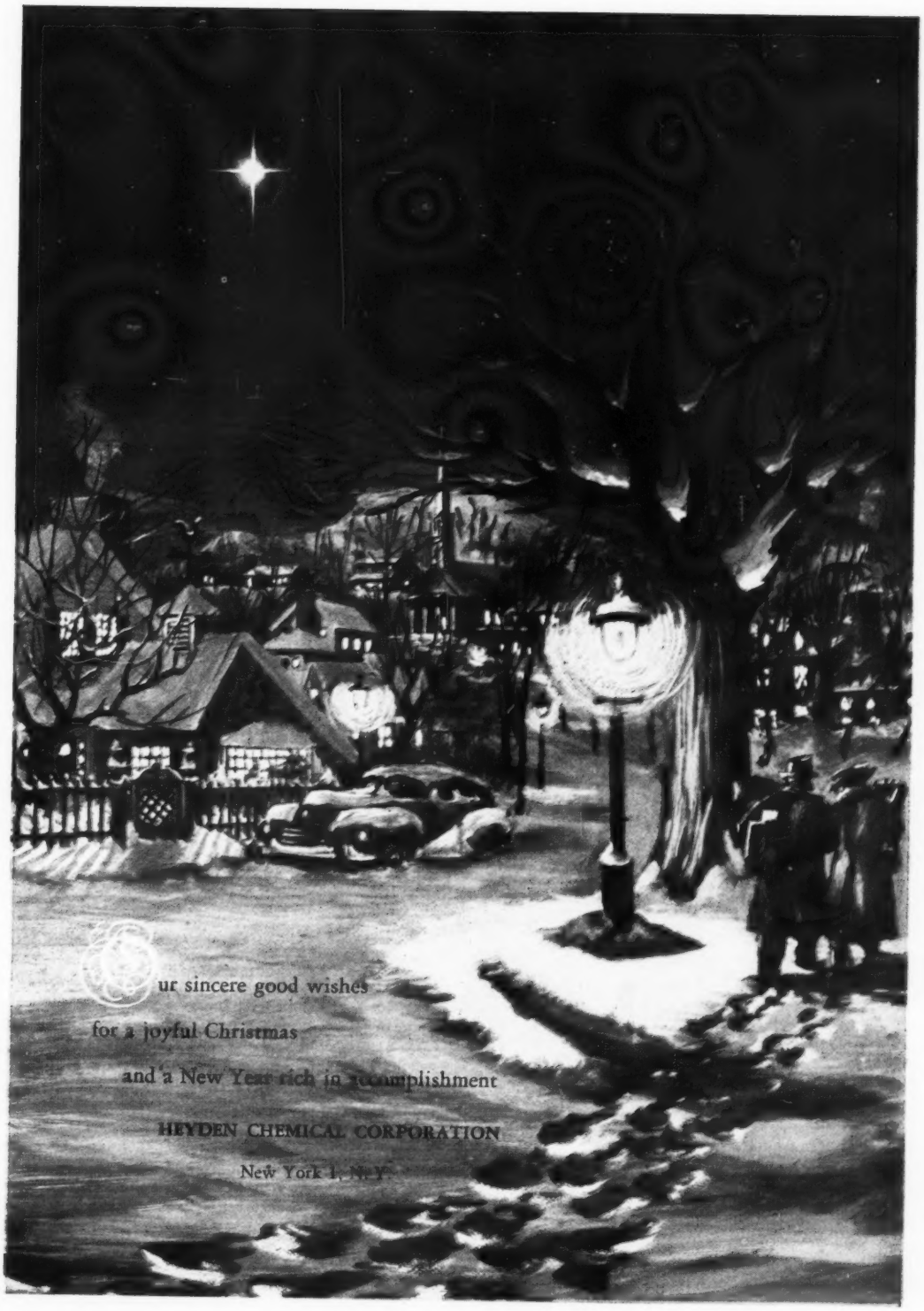
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